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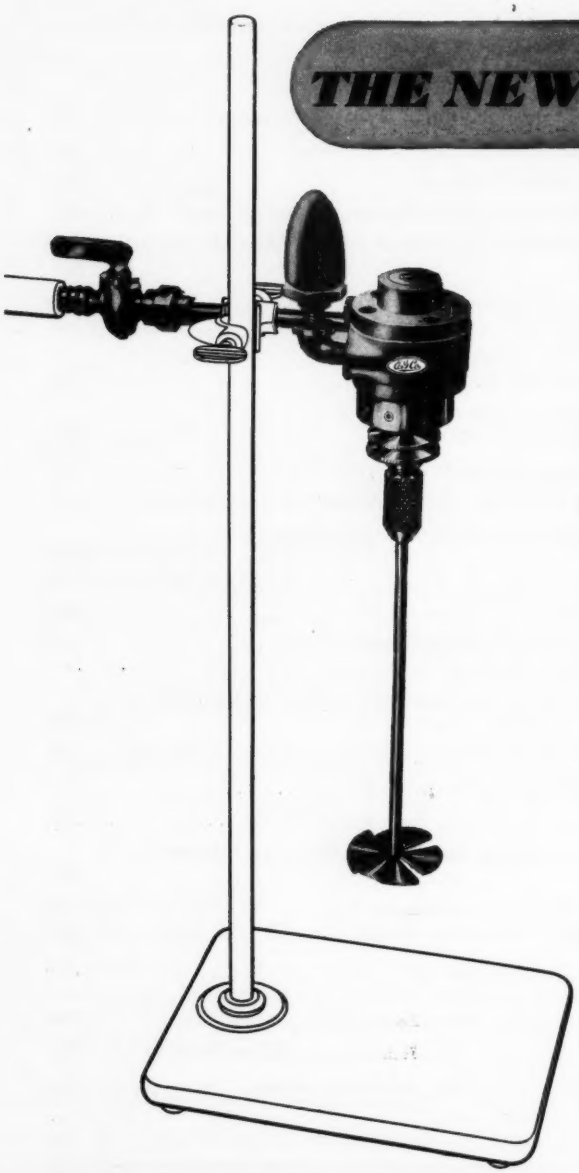

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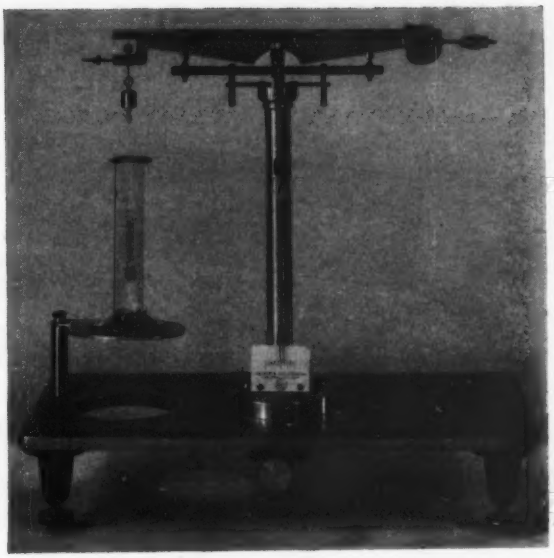

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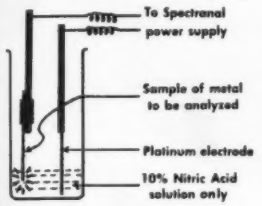

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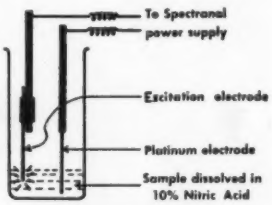

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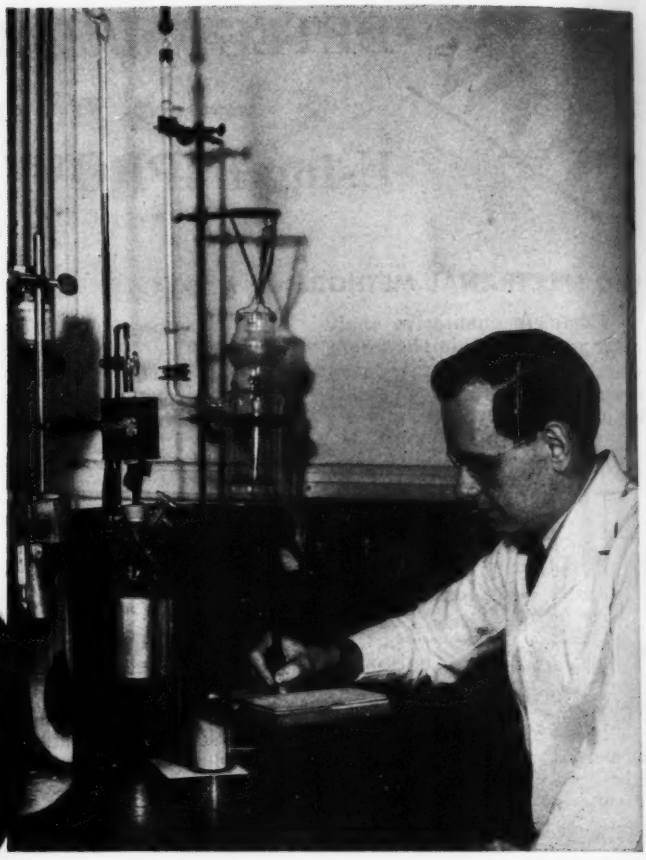

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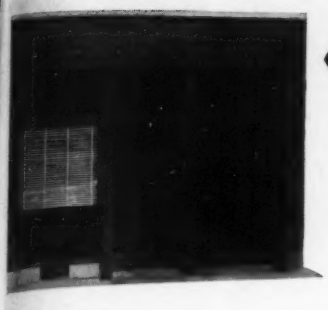

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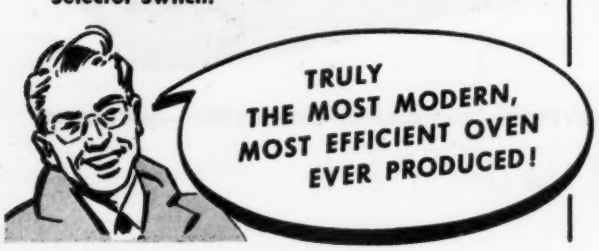

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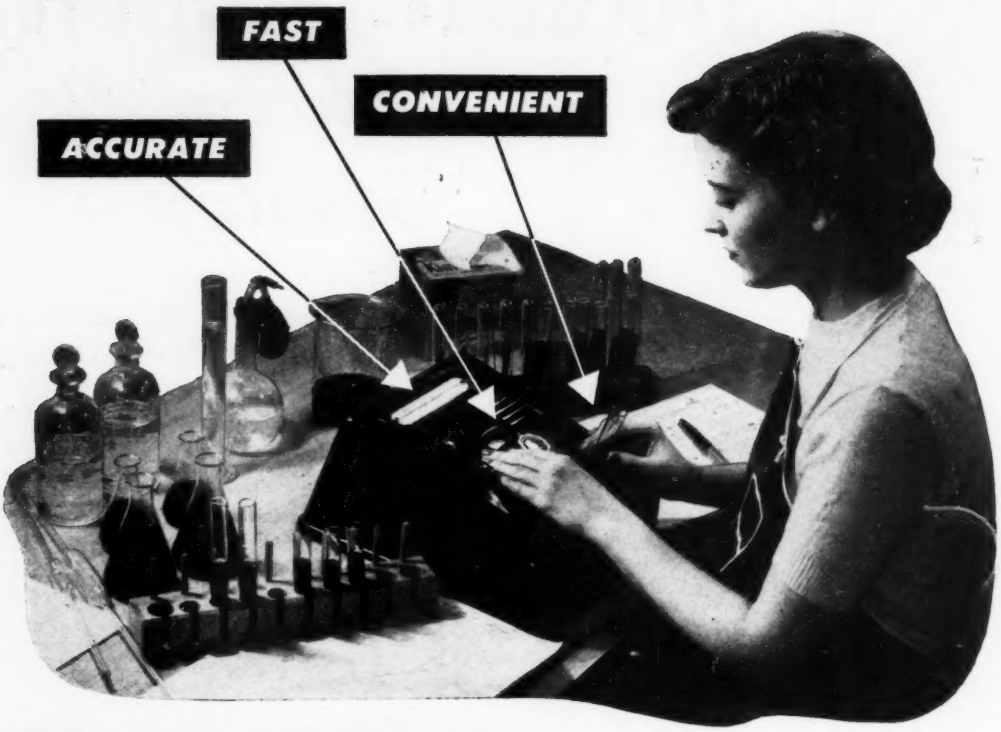

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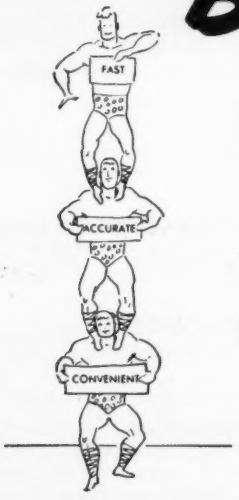

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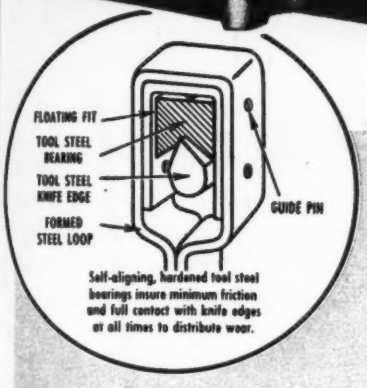

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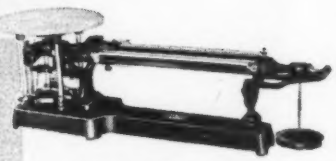

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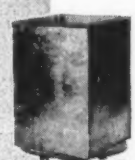

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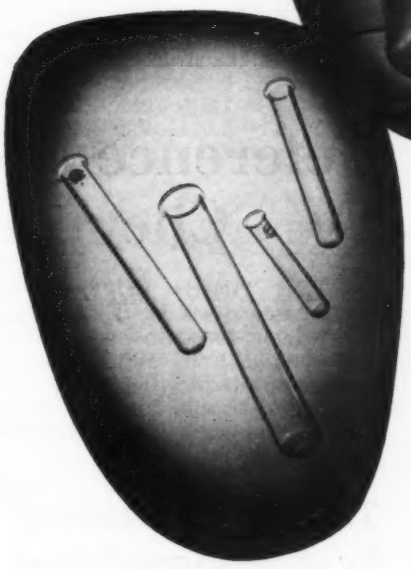

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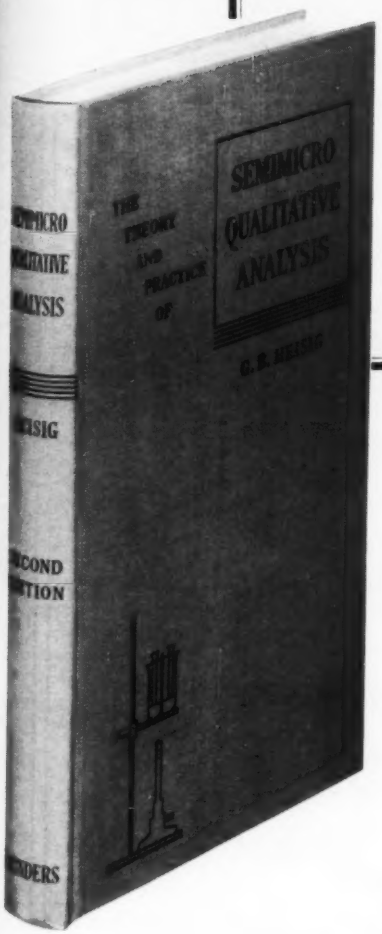

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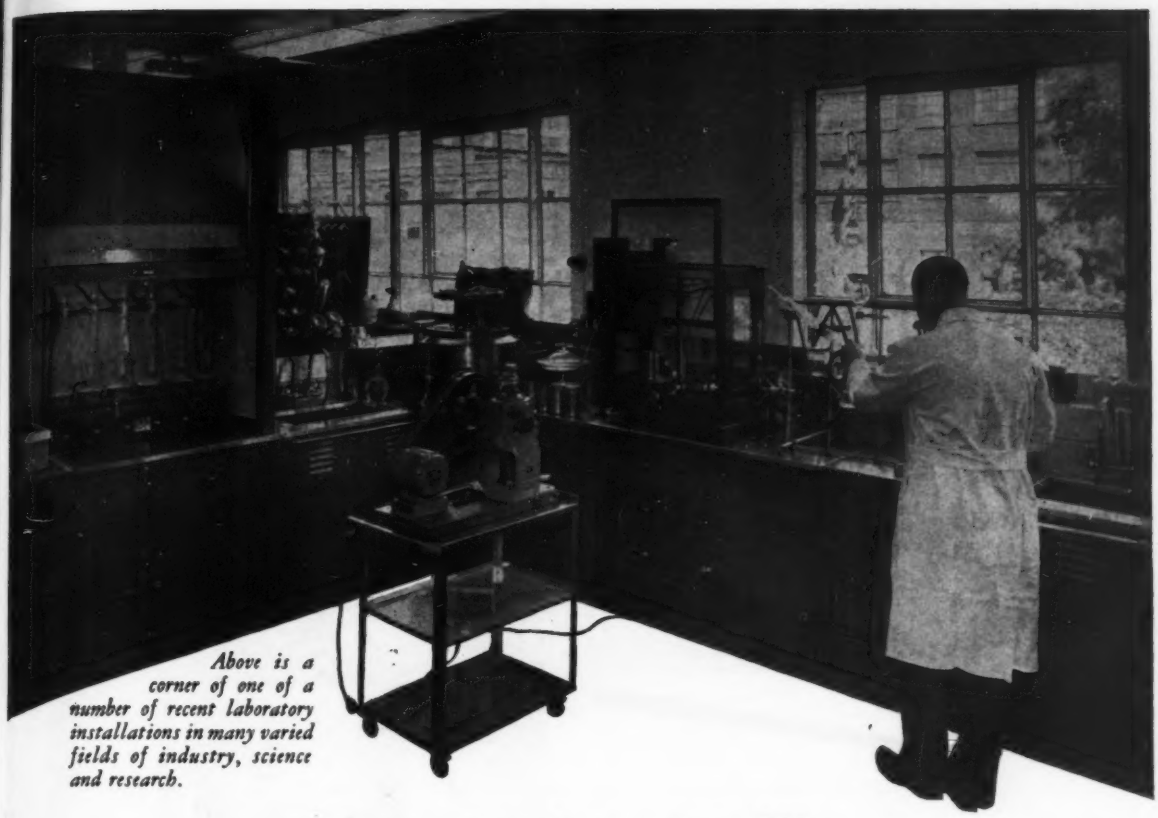

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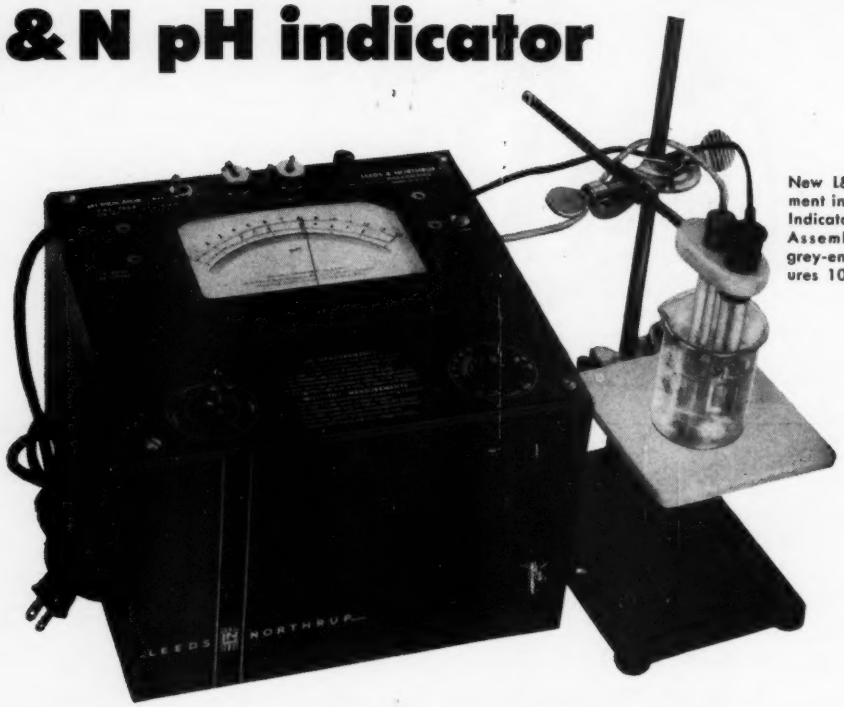

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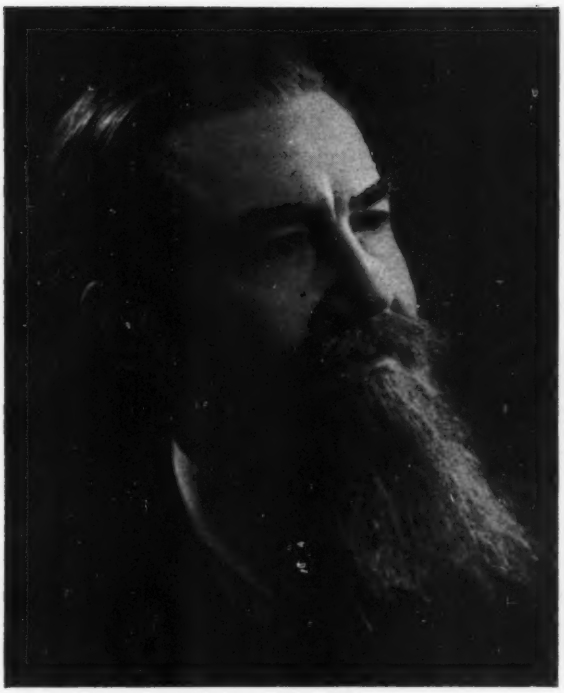

Charles Dufraisse

(See page 110)

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Editor's Outlook

FOR the first time, the Award in Chemical Education is being made at the spring meeting of the American Chemical Society this month. This award was founded by the Scientific Apparatus Makers Association, and its first recipient, Dr. Joel H. Hildebrand, was announced last fall, to receive the honor in Buffalo this spring.

A more appropriate recipient could not have been chosen to initiate the tradition of this award. THIS JOURNAL and the Division of Chemical Education have worked long and hard for the acceptance of a broad definition of the field of chemical education—a definition which would not confine it to the classroom. Such a definition has been written into the specifications for this award, and the qualifications of the first recipient are well in keeping with it. Joel Hildebrand is not only a successful teacher of chemistry—for elementary as well as graduate students—but fills practically every other qualification for distinction in the broader field of chemical education. He has written copiously and profoundly, not only textbooks but scientific monographs; he has contributed to educational methods and philosophy; he has been active in departmental and university administration; he has been a sympathetic and understanding adviser of students; he has combined research and teaching in ideal fashion and is a recognized authority in his special field; he is a leader rather than a follower in scientific circles. We are proud to have him set the standard for this award.

At the time this issue is being prepared, his medal address is not available, so we are publishing instead the paper which he read at the meeting of the American Chemical Society last fall. It represents his educational philosophy.

NOMINATIONS for the second award, to be given in 1935, have recently been called for. Any member of the American Chemical Society may make a nomination, and those interested in doing so may obtain details from the Secretary of the Society (1155 Sixteenth St., N. W., Washington, D. C.). It would seem that readers of THIS JOURNAL might be better qualified than any other group to suggest suitable candidates for this honor.

WHEN Dr. Robert Burns, the Director of the Bell Laboratories, received the Perkin Medal recently he brought up a question which is worth a good deal of consideration. Impressed with the responsibilities of the chemist in a large industrial organization and the important part he can play in determining and directing fundamental policies, he suggested that what we need is not more Ph.D.'s but more different *kinds* of chemists. His thought was that we should reserve the Ph.D. for the men who show superior talent for research but should also provide comparable types of training and experience for men with different, but equally valuable talents. According to his view this would not only be a great benefit to industry but might also encourage many more qualified students to complete their college education.

There is food for thought here. Our academic-educational structure, erected on a medieval pattern, has been badly strained by the complexity of modern civilization and industry. We have long known that its single path with its culmination "*in philosophia doctoris*" will not handle all the traffic, but perhaps we haven't done enough about it. We have, to be sure, provided some alternative types of curricula leading to other objectives and degrees, but we have tended to regard these as inferior substitutes, with the result that the intellectually ambitious have crowded onto the road to the Ph.D., which is on the way to losing its distinctive scholarly character. We need other, equally distinctive culminations to the educational road, which will be just as attractive objectives as the Ph.D. but which will recognize other types of talent. A beginning has been made in this direction, to be sure, but the whole problem has never been adequately thought out.

CHARLES DUFRAISSE

ANDRÉ ETIENNE
Collège de France, Paris, France
RALPH E. OESPER
University of Cincinnati, Cincinnati, Ohio

CHARLES ROBERT DUFRAISSE, distinguished organic chemist, was born on August 20, 1885, at Excideuil in the French province of Perigord. All of his higher training was obtained in Paris and the French capital has been the scene of his entire professional career. After obtaining the licentiate degree at the Sorbonne, he took up the study of pharmacy. In 1911 he was awarded the degree of *pharmacien de première classe* by the Faculté de Pharmacie and stayed on as assistant.

In 1917, during World War I, Charles Moureu (1863–1929), the then professor at the Collège de France, sought the collaboration of Dufraisse in the study of various products, that might have use in chemical warfare. Among these possibilities was acrolein, but this ethylenic aldehyde had the disadvantage of spontaneous resinification. Moureu and Dufraisse proved (1919–22) that this change was due to the oxygen of the air. With respect to acrolein and other similar polymerizable materials, they eventually established that these polymerizations are catalyzed by the peroxides produced in the autoxidation, and that the process can be retarded by inhibitors of autoxidation, *i. e.*, by "antioxygens." A small quantity of oxygen is capable of bringing about these polymerizations which may appear to be spontaneous, though the actual catalyst is not oxygen but a peroxide which originates in its presence.

The addition of a phenolic compound or any other known antioxygen, prevents the formation of peroxides and, therefore, stabilizes those compounds which tend to polymerize.

The discovery of antioxygens has had extremely widespread consequences involving both practice and theory; at present their application is continually increasing.

Primarily, the discovery of antioxygens provided a means of protecting numerous products sensitive to oxidation against atmospheric oxygen: rubber (aging), fats (rancidity, desiccation, spontaneous combustion), fuels (gumming), other hydrocarbons, perfumes, etc.

The first industrial application of antioxygens is due to the Americans Bierer and Davis, who used them in rubber in 1925. This application was shortly after the first publication of Moureu and Dufraisse (1921).

Another important result of the discovery of antioxygens was the proof that peroxides exert a considerable catalytic influence in promoting polymerizations. This knowledge provides the fundamental basis of the elastomer industry (Neoprene, Buna, G.R.S., etc.), which could not exist without it. It is no less important in the manufacture of several other polymeric materials (metastyrolene, polythene, etc.).

From a theoretical point of view the discovery of the antioxygen effect has had no less important consequences, especially concerning the mechanism of chemical reactions. It is sufficient to recall that the concept and the mechanism of chain reactions were first postulated in order to explain the antioxygen effect.

The research program at this laboratory was concentrated on antioxygens, until 1927, when a part of the activity was directed in another direction by the remarkable discovery of another important phenomenon, namely the reversible oxidation of rubrene, which will be discussed presently. Independently of the studies of antioxygens, Dufraisse investigated the stereochemistry of ethylenic compounds and used his findings as the basis of his doctorate thesis (1927). This same year he was appointed assistant director of the organic laboratory at the Collège de France. A little later he was named professor at the École de Physique et de Chimie Industrielles de la Ville de Paris. He divided his activity between teaching organic chemistry and his researches on photo-oxidizable molecules, which constitute another important section of his research output.

These latter studies had their beginning in his investigation of free radicals derived from acetylenic compounds. During his attempt to prepare the chloride, which he hoped would yield the diphenylphenylethynylmethyl radical, he discovered that the latter transforms into a ruby-red compound "rubrene." When shaken with air in moderate sunlight, it has the remarkable property of fixing a molecule of oxygen to form a colorless cyclic peroxide or photooxide. When heated at about 120°, the product dissociates with liberation of almost all its oxygen in the free state and regenerates the original hydrocarbon. Dufraisse named this phenomenon, which bears some resemblance to the reversible oxygenation of hemoglobin to oxyhemoglobin, reversible oxidation or *labile union of oxygen to carbon*. The intensive studies by him and his students have established the exact structure of rubrene (5, 6, 11, 12-tetraphenylnaphthacene). They subsequently showed that anthracene likewise yields a photooxide and that its mesodiphenyl derivative is the most simple acenic compound exhibiting the curious phenomenon of the labile linking of oxygen to carbon.

Dufraisse's work has included other domains. Typical instances are acrolein plastics (Orca); isomorphism; polymorphism in relation to isomerism; the photochemical formation of insoluble sulfur and its production in crystalline form; thermochemistry;

ultraviolet spectrography; etc. At present, the chief emphasis is on the study of the chromophoric properties of a new type of acetylenic and ethylenic derivative.

In 1942, Dufraisse succeeded Marcel Delépine[1] as titulary in the chair of organic chemistry at the Collège de France. The Académie des Sciences has awarded him numerous prizes and awards, and in 1948 elected him to its membership in the Chemistry Section, as successor to Lespieau. Professor Dufraisse holds the rank of *officier* in the Legion of Honor.

[1] OESPER, R. E., J. CHEM. EDUC. **27**, 567 (1950).

"SUBJECT MATTER VERSUS STUDENT"—IS IT?

JOEL H. HILDEBRAND
University of California, Berkeley, California

THE general title of this symposium seemed, when I first read it, to lend itself to at least two different interpretations. I might, of course, have written to headquarters to learn which, if either, was the one intended, but I did not wish to cramp my freedom to take a crack at both, because I could in that way best serve the purpose of a symposium of stirring up good, friendly argument.

One interpretation of the topic seemed to be this: should the teacher of chemistry be primarily concerned with his subject or with his students? This would be a special case of a general question posed during recent years by schoolmen, "Do you teach subjects or do you teach boys and girls?" Now this is a trick question, designed to justify the substitution of easy pedagogy for the rigors of subjects in the curricula of candidates for the teaching profession. It is a mere play upon words, and does not present a true antithesis. Those who ask it with any seriousness have probably never studied an inflected language, such as Latin, or German, and do not understand the distinction between the dative and accusative cases. I try to teach chemistry *to* boys and girls, I do not teach chemistry *or* boys and girls. I cannot teach chemistry to empty air, nor teach students unless they are learning something. Everyone in this audience has surely studied German, if not Latin, and well understands this distinction, but because you talk with persons who can be confused by such a question, let me give another, more homely illustration. If a farmer should say, "I feed my hogs corn," you would hardly ask him which he considers more important, feeding hogs or feeding corn. It is equally silly to consider "subjects versus children" or "chemistry versus students" as presenting valid alternatives. I trust that whoever proposed our topic did so with the hope that it would draw fire, and will not be displeased if I try to land a bullet or two on the target.

In objecting to the form of the question, I do not, however, object to the idea behind it, which is, I take it, that good teaching requires that one must not be so preoccupied with the subject as to be insensitive to the needs of the learner. Effective transmission depends both upon the receiver and upon the transmitter. But some go farther, and here I follow them with caution and even scepticism. Believing as they do, and quite rightly, that people need character more than they need knowledge, they are tempted to regard zeal for making persons good in one way or another as a substitute for professional competence. This is a subtle temptation; it sounds so altruistic; but I recommend that no one should yield to it before considering certain pitfalls. In the first place, if your victim suspects that he is to be a direct object of your uplifting zeal he is likely, if he cannot escape you, to withdraw into a shell, for few persons submit willingly to being made good. If that is your object you had better keep it dark, and get in little jabs at his character when he does not suspect you. In the second place, the objective of improving other persons' characters easily becomes trying to implant what educators call "desirable" traits, which means, bluntly put, trying to make others like themselves. That may be all right, but the effort should be accompanied by much humble soul-searching. In the third place, it is very difficult indeed to teach character, far more difficult than to teach chemistry or even arithmetic. One cannot learn how to do it by taking courses in a school of education. Character cannot be taught as subjects are taught; it does not consist of facts, formulas, and rules; it cannot be explained like proposi-

[1] Presented as part of a symposium on Subject Matter versus Student at the 120th Meeting of the American Chemical Society, New York, September, 1951.

tions in geometry. It is not something to be understood, it is to be caught, acquired, developed. The character of the teacher, if it seems admirable to the student, may influence him strongly, but what the teacher *is* far outweighs what he *says*. We need not courses *on* character but teachers *with* character—teachers who are so honest that they try first of all to do a competent job with the subject. There is no substitute for professional competence. Of course, good teaching does not stop there, it requires not only thorough knowledge of the subject but also sympathetic understanding of students. For us it should be not chemistry versus students but chemistry and students. And far from weakening the emphasis upon chemistry in the interest of strengthening character, I suggest that chemistry is a good medium for impressing several of the basic elements of good character, such as integrity, objectivity, accuracy, industry, and capacity for disinterested interest in the ways of nature.

A second possible interpretation of "subject matter versus the student" is that chemistry and the student are two parties to a conflict. Indeed, we often hear that this or that student is "struggling" with chemistry, and we see some of them obviously going down to defeat. One of mine, for example, confronted by a question in acidimetry, once wrote, "This problem could be solved by moles and equivalents, but not by me, and by the atomic weights it is too long." Another, asked to give an example of heterogeneous material wrote, "sawdust, from which the dust can be extracted by mechanical means." Still another gave this conclusion regarding air, "Air cannot be a chemical compound because chemistry was not invented till thousands of years after the creation." The distinction between an element and a compound I once learned to be as simple as this, "an element floats while a compound sinks." An example of metathesis is "A plus B squared = $A^2 + 2AB + B^2$." The chief use of CO_2, I once learned, "is to prepare H_2SO_4, champagne and other sparkling waters which do not occur free."

Such returns as these, one must admit, do sound like defeat, but they are as a matter of fact relatively rare, and not characteristic of a class as a whole. Such answers come from victims of poor high-school training; or of parental ambition outrunning the ambition of the student; or of such preoccupation with what are called "student activities" that studies are inactivities; or of poor choice of ancestors, like the student whose mother wrote, "Mr. X is willing to do his work and is putting forth every efford. Due to serious headaches in his head that causes his blood to rush faster than usual to his head, and it keeps him from thinking as he should."

The large majority of students in my freshman classes do not display antagonism to the subject. For most it is a recognized part of the foundation for their intended occupations. For some it is the science in a liberal arts curriculum, deliberately elected from a variety of possibilities and studied with eager curiosity. A not negligible number arrive in the university already fascinated by the subject through experience with home chemical sets or exposure to superior teaching in high school. And finally, if these incentives have been weak, what other subject of freshman study affords the lecturer such opportunities to make the subject interesting as does chemistry—with the variety of colors, odors, explosions, and volcanoes at his command, as well as other experiments, less dramatic, perhaps, but more thought provoking. Of course, if they are to be effective they must not very often fail. One must not have to offer an excuse like the one made too frequently by a certain lecturer, "Well, gentlemen, the experiment is a failure but the principle holds just the same."

But the conflict metaphor is inappropriate for another reason; chemistry is not a party to a struggle; it does not attack the student. A better metaphor would be that it is intellectual food, intended to nourish and strengthen him—even if rather rich, it must be confessed, for weak digestions. Or, it presents a succession of hills and mountains for him to climb, if he has the necessary stamina and determination. But there are no trenches on the slopes filled with enemy troops to oppose his ascent. Each of these eminences affords an interesting purview to those who surmount it. Both the view from the top and the satisfaction of being able to achieve it furnish motives for the ascent. The more difficult the climb, the greater is the satisfaction in being able to make it.

In my long observation of student motives, attitudes, and reactions, I have come to the conclusion that where a sense of hostility exists it is more often between student and teacher than between student and subject, and even where the latter exists it arises all too often from a spread of distaste from instructor to subject. I ask your permission, therefore, to alter the topic to "instructor versus student" and see what we can make of it. Where that conflict is felt by either party, it is easy for each to blame the other, and that does not get one very far. I propose, therefore, that we at least begin by giving the student the benefit of the doubt and consider what grounds a student may have for the antagonism which he may feel towards his instructor. Such antagonism would not be so serious if the instructor were the only loser, but it is a grave matter for an instructor to get between student and subject and spoil it for him. To realize that this is often the case we have only to recall our own experiences as students. I know of one student for whom the beautiful subject of organic chemistry was long made distasteful by being presented only as formulas and recipes, melting and boiling points, copied out by the instructor from a textbook the hour before the lecture and then retailed for students to copy in their turn. It would have been far better if that particular instructor had taken himself out of the way and let students study directly from the book, in their own rooms, in comfortable chairs. The moral of this case is that most knowledge is available in adequate books, and a teacher who merely recites from the book is wasting students' time. The author of the

book may be a better man than the instructor, and it should be taken for granted that students can read. If the teacher has no effective supplement to the book, he should change his job.

Every lecturer would do well, in my opinion, to maintain a sceptical attitude towards his own lectures. I have almost always led a quiz and laboratory section as a post-mortem on my own lectures, and many has been the time that I have discovered by talking with students that what I supposed I had made crystal clear in lecture was about as clear to them as a San Francisco fog. In the absence of such appraisal, the lecturer may easily think his lectures much better than they really are. It has been ironically remarked that the two chief advantages of lecturing are, first, that it helps the professor to develop a fine, confident personality because he never hears criticism and, second, that it enables the student to get an education without ever having to learn to read.

I must not neglect to mention another, even simpler method of appraising one's teaching, which is to look into the faces of the class for signs that their minds are in operation. A teacher has ample opportunity, from the responsiveness of his class, from informal quizzing, and from friendly conversation with individual students, to appraise the effectiveness of his teaching. He needs no questionnaires circulated at the instance of administrative officers. Such questionnaires constitute an unwarranted interference with the delicate, difficult task of establishing friendly relations between student and instructor which I would deeply resent; they suggest that the instructor may not be fair, or interesting, or competent, or master of his subject. The administration which has to learn these things from students should have its own competence and fairness appraised by a questionnaire prepared by the faculty. There are dozens of ways to improve teaching,[2] but the anonymous, administrative questionnaire is not one of them. No man who is dull, humorless, or pedantic is to be scared by questionnaire returns into becoming suddenly brilliant, stimulating, and sympathetic.

This reminds me to state that I have a plan for determining the proper time for the retirement of a professor. It is a substitute for arbitrary retirement at some uniform age, 65, 67, or 70, as the case may be. Professors are supposed by trustees to become suddenly incapacitated at some such age, although trustees, of course, remain just as good after these ages as they were before. My plan includes, first of all, a surreptitious count of the fraction of his classes still awake at 15 minutes before the end of the period; second the same criteria of scholarly productivity as were applied upon promotion to the full professorship; third, secret testimony from colleagues regarding the candle power of his conversation at the faculty club lunch table; and finally, evidence of ability to ski, climb mountains, or dive from the 12-foot springboard. (I put this last in for my own benefit, to help out in case I should be adjudged weak on any of the other counts.) I think my plan is so good that it might well be applied to professors long before the age of 65. I am confident that it would diminish the instances of conflict between student and instructor. Indeed, I think so well of it that I recommend a corresponding plan for self-application by boards of trustees.

Now if you will continue to think of your own student days, you will recall, I doubt not, irritation over the habit of some teachers of trying to appear to know all the answers. They tell in advance how every lecture experiment is going to turn out, thus entirely spoiling it for the student as an experiment. They try to arouse admiration for themselves instead of curiosity about the ways of nature. They fail to give students a true impression of the unexplored areas of science, and of the dynamic, tentative nature of scientific endeavor.

Another source of student disgust is failure to be considerate and courteous, abuse of authority, enjoyment of the sense of power derived from having students at one's mercy. It is all too easy to become exasperated, to assume that dullness in the student is voluntary. Sometimes, indeed, it is, but not ordinarily. We easily forget that chemistry is indeed difficult, and that even we, ourselves, once had some trouble with it, and that we did not respect instructors who acted toward us like "schoolmarms."

Again, nothing is more effective in maintaining the good will of the student than the utmost fairness in grading. He should feel convinced that the same standards are being applied to every member of the class, and that his grades do not depend upon his good or bad fortune in the assignment of section leaders. I could say a good deal about machinery for attaining this end.

I think it helps also to recognize the great spread of preparation, interest, and ability in a freshman class and to arrange both instruction and grading so that the brightest and well prepared are stimulated to do their best, while the slower and more poorly prepared are not disheartened by too many tasks far beyond their capacity. This also calls for ingenuity on the part of the one in charge of the course, but it can be done.

But now, in conclusion, I do not wish to be lecturing you, my professional colleagues, like a schoolmarm myself, nor do I wish to appear peevish about the title, "Subject Matter versus Students." I have had my fun with it, deliberately giving it my own interpretations. There is a third interpretation, one to which we doubtless all subscribe, which, I take it, may be paraphrased somewhat as follows: our task is not to stuff subject matter down the throats of students for them to regurgitate to us on demand, but rather our privilege is to develop in each student whatever capacity he may have for clear thinking and for appreciating and understanding something of the great science of chemistry. Anything that we can add as a side line in the way of character development is all to the good. Teaching so as to achieve such aims, however imperfectly, is a high calling, and one which is worthy of all the skill and devotion we can bring to bear upon it.

[2] *Bull. Am. Assoc. Univ. Prof.*, 5, 638 (1945).

ENZYMES, THE BASIS OF LIFE

JAMES B. SUMNER
Cornell University, Ithaca, New York

THE digestive action of gastric juice was known to de Réamur and to Spallanzani, but the first definite enzyme discovered was possibly the enzyme in plant roots that was observed by Planche in 1810 to turn tincture of gum guaiac blue, or else the amylase, or starch-digesting enzyme of wheat, found by Kirchoff in 1814. After this time other enzymes were discovered and by 1900 it was clearly understood that enzymes were necessary for digestion and that other enzymes, present in living cells, catalyzed various other chemical reactions. However, the tremendous importance of enzymes for life has come to be fully appreciated only during the past 20 years. The investigation of enzymes and enzymic reactions has already partly explained such phenomena as photosynthesis, nitrogen fixation, muscular contraction, nervous conduction, growth, disease, and the production of cold light. At the present time the chemist, physiologist, bacteriologist, and medical scientist are all interested in enzymes. The up-to-date bacteriologist is an enzyme chemist pure and simple and the organic chemist who is not well informed about enzymic reactions is behind the times. A very rapid development is now taking place in this field.

The ability of any organism to carry on and also to adapt itself is truly remarkable. It can digest foods, detoxicate poisons, and synthesize complicated hydrocarbons, carbohydrates, lipids, and proteins, as as well as dyes, drugs, perfumes, alkaloids, and toxins.

It has been found that the enzymes of various species are frequently similar and that the reactions catalized by these enzymes are identical. Thus glycogen is broken down step by step in both animals and in yeast cells in practically the same way until the last steps are reached. In yeast the products formed are largely alcohol and carbon dioxide, in animals water and carbon dioxide. Now why is there this similarity? Is it because all living things, in spite of their diverse outward appearances, are related, or is it because the chemical reactions required for life are limited in number?

Enzymes are significant because they are the agents responsible for life. They are what makes the living thing tick. Life without enzymes would be impossible.

Genes are believed to bring about the synthesis of enzymes, and for every enzyme some four or five different genes are believed to be necessary. Enzymes are sometimes observed to be identical with toxins, but some toxins have not been shown to exert enzyme action. Tetanus toxin, now known to be a crystallizable protein, is so poisonous that 1 mg. would suffice to cause the death of 100 tons of guinea pigs. It is difficult to conceive of a chemical reaction that could be the cause of this toxicity. Since toxins are destroyed by heating, well-cooked tetanus toxin probably would be a palatable food.

Hormones are thought to function by modifying the action of enzymes. Vitamins are employed by cells as materials for the synthesis of certain enzymes, usually oxidizing enzymes. Viruses certainly are not enzymes, although some viruses contain enzymes.

We now realize that protoplasm is a mixture of enzymes. These enzymes are not distributed at random in the cell, but are localized in certain structures. Some enzymes are found in the cytoplasm, others in the nucleus. One enzyme, lecithin phosphatase, is present exclusively in the nucleolus. One group of enzymes, the Krebs cycle enzymes, occurs in the mitochondria. Cells which excrete enzymes produce granules in the vicinity of the Golgi apparatus and these granules are thought to be composed of these particular enzymes.

PROPERTIES OF ENZYMES

The author (*1*) first showed in 1926 that the enzyme urease was a crystallizable protein, and Northrop (*2*) crystallized pepsin in 1930. Since then many other enzymes have been obtained in pure and crystalline condition and have been shown to be proteins.

Urease is probably the easiest of any enzyme to crystallize. Stir 100 g. of jack bean meal with 500 ml. of 32 per cent acetone at 28°C., pour upon a filter, and place in the ice chest. The next day the filtrate will be found to contain microscopic urease crystals. Urease can be tested quantitatively by allowing an aliquot of properly diluted urease solution to act upon urea in neutral phosphate at 20°C. at pH 7.0 for five minutes. The ammonia formed is then determined quantitatively.

Enzymes are usually colorless, but may be yellow, green, blue, or greenish brown. Most enzymes are soluble in water or dilute salt solutions, but some occur attached to insoluble portions of cells and either cannot be made to dissolve or else can be dissolved only by an unusual procedure. Diaphorase is an example of the latter type. Straub (*3*) was able to prepare solutions of this enzyme by heating the suspended material (washed heart tissue) to 43°C. in a solution of dilute ethyl alcohol and ammonium sulfate at pH 4.6.

The catalytic activity of some enzymes is very great. For example, one molecule of beef-liver catalase will decompose five million molecules of hydrogen peroxide every minute at 0°C. This is called the "turnover number." Blood catalases are still more active than liver catalase. One can never be dogmatic about enzymes. Some are rather inactive. The

"old yellow enzyme" has a turnover number of only 50 and *l*-amino acid oxidase (*4*) of rat kidney has a turnover number of only 7.

Most enzymes are very unstable and are destroyed upon heating to 80°C. Lipoxidase in dilute solutions is totally destroyed upon shaking gently in a test tube for five minutes. However, a solution of peroxidase kept by the author nine years in an ice chest was found to be very active. A catalase solution dried in an oven at 80°C. for 24 hours was very active.

One of the most striking and characteristic properties of enzymes is their specificity. We find different varieties of specificity. Thus urease is absolutely specific. Of all of the hundreds of substrates which have been tested urea is the only one which is hydrolyzed by it. Recently Shaw and Kistiakowsky (*5*) claimed that urease could hydrolyze biuret. In a later publication they admitted that their biuret contained urea.

Some enzymes are relatively specific. Peroxidase is an enzyme in this class. It catalyzes the oxidation of an indefinite number of phenols and aromatic amines in the presence of hydrogen peroxide or other peroxides.

One of the least specific enzymes is the xanthine oxidase of milk. This enzyme catalyzes the oxidation of alcohols, purines, and also reduced coenzyme I.

Enzymes exhibit stereochemical specificity. As an example of this sort of specificity, the β-glucosidase of sweet almonds will catalyze the hydrolysis of an indefinite number of β-glucosides, but does not affect α-glucosides. α-Glucosides are hydrolyzed by the α-glucosidase of yeast. *d*-Amino acid oxidase brings about the oxidation of *d*-amino acids only, while *l*-amino acid oxidase acts upon *l*-amino acids.

COENZYMES

It has been mentioned previously that enzymes are either simple proteins or else conjugated proteins. Those that are conjugated proteins are composed of a protein part united with a prosthetic group of low molecular weight. This prosthetic group, known as the coenzyme, is composed of some vitamin, modified by the addition of phosphoric acid or phosphoric acid plus certain organic groups. It is evident that we eat vitamins in order to obtain material necessary for the synthesis of enzymes.

The affinity of the coenzymes to the protein part of the enzyme (apoenzyme) varies considerably. In diphosphoglyceric aldehyde dehydrogenase the coenzyme, coenzyme I, is loosely bound. This is of advantage in the production of ethyl alcohol by yeast. Here the diphosphoglyceric aldehyde gives up hydrogen, and this is taken up by the coenzyme. The coenzyme then migrates off and unites with the protein part of alcohol dehydrogenase. This enzyme now gives up its hydrogen to acetaldehyde, thereby forming ethyl alcohol.

Warburg and Christian (*6*) succeeded in splitting the "old yellow enzyme" into its prosthetic group, riboflavin phosphate, and its protein part.

Their second and most satisfactory method consisted in adding 12.5 ml. of saturated ammonium sulfate to 12.5 ml. of enzyme solution and then adding with stirring 4.4 ml. of 0.1 *N* hydrochloric acid. The precipitated apoenzyme was centrifuged down, washed once at 0°C. with 25 ml. of half-saturated ammonium sulfate, dissolved in 2 ml. of 0.5 *M* phosphate of pH 7.4 at 0°C., and diluted to 12.5 ml. The mother liquor, containing riboflavin phosphate, was brought to pH 7.4 by the addition of 0.5 *M* dibasic phosphate. When the solution of the apoenzyme was mixed with the solution of riboflavin phosphate the old yellow enzyme was reconstituted.

Warburg and Christian (*6*) took the apoenzyme part of the "old yellow enzyme" and by adding flavine adenine dinucleotide to this were able to synthesize an entirely new enzyme. The properties of this new enzyme were the same as those of the "old yellow enzyme."

Warburg and Christian (*7*) split *d*-amino acid oxidase into its apoenzyme and its prosthetic group, flavine adenine dinucleotide, or FAD. The enzyme can be reconstituted by adding these two parts. The apoenzyme of *d*-amino acid oxidase can be kept indefinitely in the dry state in the ice chest and is of value if one wishes to test for small quantities of FAD. Here one adds the solution suspected of containing FAD to a solution containing 1 mg. of the apoenzyme in pyrophosphate buffer. One next adds *d*-alanine and incubates. If FAD was present the *d*-alanine will be oxidized to pyruvic acid and ammonia.

Theorell (*8*) was able to split purified horse-radish peroxidase into its protein part and hematin and to resynthesize peroxidase by adding hematin to the protein. Gjessing and Sumner (*9*) have attempted to synthesize various peroxidases by adding to the apoenzyme of horse-radish peroxidase the hematin part after replacement of the iron atom of the hematin with such metals as cobalt, manganese, copper, and nickel. They found that the compound of the apoenzyme with manganous protoporphyrin had some activity, and that compounds of the apoenzyme with mesohematin and hematohematin also possessed activity. Theorell (*10*) found that the mesohematin compound possessed activity but that the other compounds did not. In a recent article Maehly (*11*) compares the activity of three synthetic peroxidases with the activity of ordinary (protohematin) peroxidase. This is shown in the following table.

Prosthetic group	*Percentage activity*
Protohematin	100%
Deuterohematin	63%
Mesohematin	57%
Azahematin	20%

THE MECHANISM OF ENZYME ACTIVITY

An enzyme does not act by its mere presence and thus is not similar to the philosophers' stone, which was assumed by the alchemists to be capable of transmuting base metals into gold. Michaelis and Menten were among the first who proposed that the enzyme

forms an intermediate compound with the substrate and that this compound then decomposes into the products and the original enzyme. In 1941 Sumner and Dounce (*12*) proposed that the enzyme catalase reacts with hydrogen peroxide to form a peroxide of catalase and that this peroxide then reacts with a second molecule of hydrogen peroxide, regenerating the catalase:

$$\text{Cat.OH} + \text{H}_2\text{O}_2 = \text{Cat}\cdot\text{OOH} + \text{H}_2\text{O}$$
$$\text{Cat.OOH} + \text{H}_2\text{O}_2 = \text{Cat}\cdot\text{OH} + \text{H}_2\text{O} + \text{O}_2$$

The work of Chance (*13*) has proved conclusively that this hypothesis is basically correct.

In the introduction to his book, "The Hydrogen-transferring Enzymes," Warburg (*14*) makes the statement that the most important task confronting the enzyme chemist is to explain why an enzyme acquires activity when the protein part unites with the prosthetic group. It would appear likely that the activity of an enzyme possessing a prosthetic group is connected somehow with a linkage formed between the substrate and both the prosthetic group and the protein part of the enzyme. It is probable that the specificity of an enzyme is the result of a uniting of the substrate with the protein part of the enzyme.

A large number of enzymes have been found to require metallic ions for their catalytic activity. Thus, arginase requires manganous, cobaltous, or other divalent ions; phosphoglucomutase requires magnesium, manganous or cobaltous ions; most of the phosphatases require magnesium ions and so do the peptidases. E. L. Smith (*15*) and others have postulated theories to explain how these metallic ions might function by forming linkages between the enzyme and its substrate. These explanations doubtless are very suggestive but they have not yet explained the whole story.

Oxidation brought about by enzymes has been described as an addition of oxygen, as a loss of hydrogen, or as a loss of electrons. Certain enzymes are spoken of as oxidases and others as dehydrogenases. Glucose oxidase, otherwise known as notatin, is an enzyme present in molds and is ordinarily considered to be an aerobic oxidase. It oxidases *d*-glucose to gluconic acid and hydrogen peroxide if gaseous oxygen is present. Bentley and Neuberger (*16*) have postulated that if this enzyme adds one atom of oxygen to the glucose and one to water containing heavy oxygen the reaction will be:

$$\text{C}_6\text{H}_{12}\text{O}_6{}^{16} + \text{O}_2{}^{16} + \text{H}_2\text{O}^{18} = \text{C}_6\text{H}_{12}\text{O}_7 + \text{H}_2\text{O}^{16}\text{O}^{18}$$

In this case hydrogen peroxide containing heavy oxygen would be produced. This was found not to occur. If glucose oxidase causes a dehydrogenation the reaction will be:

$$\text{C}_6\text{H}_{12}\text{O}_6{}^{16} + \text{O}_2{}^{16} + \text{H}_2\text{O}^{18} = \text{C}_6\text{H}_{10}\text{O}_6{}^{16} + \text{H}_2\text{O}_2{}^{16} + \text{H}_2\text{O}^{18}$$

Here the hydrogen peroxide will contain ordinary oxygen. This was found to be true. Therefore, glucose oxidase is a dehydrogenase. It splits two atoms of hydrogen from the glucose molecule, forming the δ-lactone of gluconic acid which later hydrates to gluconic acid. The two atoms of hydrogen go to the isoalloxazine ring present in the glucose oxidase; next this reduced enzyme reacts with gaseous oxygen and forms hydrogen peroxide.

INFLUENCE OF THE NUTRITIONAL STATE

Well-nourished animals contain greater enzyme concentrations than starving animals. In some cases an enzyme may be absent in a young animal. Thus, Westerfeld and Reichert (*17*) showed that young rats contain no xanthine oxidase in their livers, but rapidly develop this on a diet rich in liver, cream, and protein. Some unknown substance that occurs in cream and liver is necessary.

SYNTHESIS WITH ENZYMES

For many years the synthesis of starch, cane sugar, fats, proteins, and hundreds of other compounds was looked upon as a mysterious process that required living cells. The best explanation offered was that the synthetic reaction was catalyzed by the same enzyme that could cause a breakdown or a hydrolysis and that a reversal of the hydrolysis was somehow brought about by a removal of water. Every chemist is familiar with the expression:

$$\frac{(\text{C}_2\text{H}_5\text{OH})(\text{CH}_3\text{COOH})}{(\text{CH}_3\text{COOC}_2\text{H}_5)(\text{H}_2\text{O})} = K$$

Here K is the equilibrium constant and its value is about 0.25. If water is added to this system more acetic acid and alcohol are formed. If water is removed more ethyl acetate is formed. The enzyme catalyzing this reversible reaction is esterase. Bourquelot and Bridel (*18*) were able to synthesize a number of β-glucosides using the β-glucosidase of sweet almonds and allowing it to act upon *d*-glucose and various phenols. Here the concentration of water was diminished by employing alcohol in a concentration of 30 per cent, or more.

In living cells synthesis is probably brought about in a different way. In some instances a compound is hydrolyzed by one route and is synthesized by an entirely different route. Thus, urea is hydrolyzed to ammonium carbonate by the enzyme urease. It is produced together with ornithine by the action of arginase upon arginine. Later on, enzymes in the liver convert the ornithine into citrulline and then into arginine, which is again acted on by arginase.

Enzymic reactions can be divided into (1) the type which proceeds to completion, and (2) the type which goes to a point of equilibrium where both substrates and products are present in measurable amounts. As examples of the first type we have:

$$2\text{H}_2\text{O}_2 = 2\text{H}_2\text{O} + \text{O}_2$$

Here the enzyme is catalase. After this enzyme has acted for a short time there will remain no detectable quantity of hydrogen peroxide. Another reaction of this sort is:

$$HCN + Na_2S_2O_3 = NaSCN + NaHSO_3$$

This reaction is catalyzed by rhodanese, present in liver. Here again we find that after a short time no trace of hydrocyanic acid remains. Teleologically it is important to have both hydrogen peroxide and hydrocyanic acid completely removed, since they are very poisonous. A third reaction of this type is:

$$\text{sucrose} \rightarrow \text{gum dextran} + \text{fructose}$$

This reaction is synthetic, since gum dextran has a molecular weight of 1 to 2 millions. The reaction goes to completion. By testing immunologically for the gum dextran it is possible, according to Hehre (*19*), to detect the presence in the original solution of 1 part of sucrose in 100,000 parts of water.

An example of the second type of reaction is:

$$\text{glucose-6-phosphate} \rightleftharpoons \text{fructose-6-phosphate}$$

This reaction is catalyzed by phosphohexose isomerase. At the point of equilibrium there is 70 per cent of glucose-6-phosphate and 30 per cent of fructose-6-phosphate.

Another reaction of this second type is:

$$\text{glucose-1-phosphate} \rightleftharpoons \text{starch amylose} + \text{phosphate}$$

This reaction is catalyzed by phosphorylase. At equilibrium at pH 7.0, using rabbit muscle phosphorylase, the Coris (*20*) found the concentration of glucose-1-phosphate to be 23 per cent and the concentration of phosphate 77 per cent. The reaction will not go from left to right unless a trace of glycogen or amylopectin is present. It should be pointed out that the reaction here is a phosphorolysis and not a hydrolysis.

One more type of reaction remains to be discussed. This is the endergonic type, as contrasted with the preceding types which are spoken of as exergonic. In the endergonic reaction energy must be supplied. This comes from the breakdown of adenosine triphosphate, or ATP. Thus, the synthesis of glucose-6-phosphate, which is catalyzed by hexokinase is:

$$\text{glucose} + \text{ATP} = \text{glucose-6-phosphate} + \text{ATP}$$

By employment of ATP, or systems which form ATP, biochemists have synthesized urea, glutamine (from glutamic acid), acetyl choline, and compounds with peptide bonds.

A number of years ago Vestin (*21*) synthesized coenzyme II by incubating coenzyme I with ATP and yeast juice. More recently coenzyme I, riboflavin phosphate, and flavin adenine dinucleotide have been synthesized by the action of enzymes.

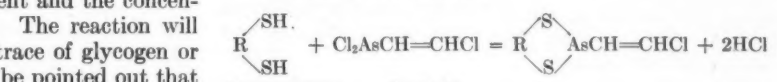
THE ACTION OF HORMONES UPON ENZYMES

The hormones are doubtless produced by the action of enzymes and likewise are destroyed by enzymes. The question which confronts us here is how hormones exert their action upon the animal and why an extremely minute amount of hormone can be so effective. It has been demonstrated that the administration of a hormone can alter the concentration of an enzyme in an organ but this change in concentration by no means explains the characteristic effect of the hormone. At a recent symposium of the New York Academy of Sciences (*22*) much experimental evidence was offered and many theories were described, but no satisfactory explanations were reached.

POISONS

When an animal, plant, or microorganism is poisoned the effect is due mainly to the action of the poison upon the enzymes. Medicines, which usually are to be classified as poisons, exert their effect by inactivating either the enzymes of the host or else the enzymes of the invading microorganism or parasite. In the case of poisoning by cyanide, some of the enzymes that are inactivated are catalase, the peroxidases, and cytochrome oxidase. Iodoacetate inactivates phosphoglyceric aldehyde dehydrogenase, while fluoride inactivates enolase. The choline esterases are inactivated by such poisons as prostigmine, eserine (an alkaloid from the Calabar bean), and by tri-*o*-cresyl phosphate, diisopropyl fluorophosphate, ethyl paranitrophenyl thionobenzene phosphate and tetraethyl pyrophosphate. Curare and quinine also inactivate the choline esterases.

Lewisite, the "war gas," is believed to derive its destructive effects by reacting with enzymes which contain sulfhydryl groups:



tissue enzyme Lewisite

The antidote for Lewisite is British Anti-Lewisite or 2,3-dimercaptopropanol, which replaces Lewisite, setting the enzymes free. Enzymes that contain SH-groups unite readily with mercuric, silver, lead, and other metallic ions.

Hundreds of other examples of the inactivation of enzymes by poisons are known.

When enzymes are present in the wrong place they can act as poisons. Thus, a milligram of crystalline trypsin injected intravenously into a rat is said to cause death in about four minutes. If papain is injected into the circulation it will cause death by intravascular coagulation. One-seventeenth of a mg. of crystalline urease will kill a 2-kg. rabbit within a few hours by producing poisonous ammonium salts from the rabbit's urea. Snake venom has been shown by Zeller (*23*) and others to be a concentrated solution of various enzymes. When the venom is introduced into the body it immediately begins to tear the tissues apart by hydrolysis and oxidation. The animal simply disintegrates. Scorpion and bee venoms are similar to snake venoms, but smaller quantities are injected by these insects. Scorpion bites do not kill adults but often are fatal to children under five years of age. One of the harmful enzymes in venom and present also in bacteria is lecithinase A. This decomposes the lecithin in the body into lysolecithin. The lysole-

cithin thus formed destroys the erythrocytes. Another enzyme in venom is ATPase. This destroys the ATP which is necessary as a primary source of energy.

Bacteria often contain the enzyme hyaluronidase. This digests the hyaluronic acid present in the skin and tissues and allows the bacteria to spread.[1] Hyaluronidase is present in spermatozoa where it performs the useful function of digesting away cumulus cells surrounding the ova and allowing fertilization to occur.

ENZYMES AND FOODS

Foods can lose their flavor because their volatile constituents are allowed to evaporate (keep coffee in the deep freeze) or can lose their color when exposed to sunlight (fugitive coloring matters such as anthocyanins), but the important changes that are likely to happen to foods are brought about by enzymes. These enzymes may belong to the foods themselves or may be introduced in the bacteria, yeasts, molds, insects, rats, or mice that have access to the foods.

As an example of the spoilage of foods by enzymes already present, mention can be made of the lobster, which after death quickly undergoes self-digestion, or autolysis, and must therefore be kept alive until ready to be boiled. Bananas will ripen by the action of their own enzymes and finally become over-ripe and worthless. Potatoes that have been cut into slices turn brown and then black, as every housewife knows. The enzymes that cause this oxidation are laccase and tyrosinase.

When there is bacterial contamination of foods some of the products formed by enzymes can cause serious poisoning. Yeasts can be used to preserve foods. Thus, the natives in both Africa and South America chew starchy grains or roots and spit out the chewed material into a container of bark. The saliva contains amylase, which digests the starch. This forms maltose. Yeasts fall into this material and bring about alcoholic fermentation. The alcohol thus formed is thought to have a preservative effect. Later the material is eaten.

There are three procedures which can be employed to prevent the destruction of foods by the action of enzymes. These are heating, which usually destroys the enzymes, drying, which inactivates the enzymes, and cooling to low temperatures, which greatly diminishes the action of enzymes.

Enzymes are responsible for the synthesis of vitamins and can also destroy vitamins. Thus, the lipoxidase in soybeans oxidizes and destroys vitamins A and the carotenes (provitamin A). Ascorbase, present in many vegetables, destroys ascorbic acid. Thiaminase, present in certain fresh-water fish, destroys vitamin B_1, or thiamine.

Penicillin, which is an antibiotic and not a vitamin, is destroyed by penicillinase, present in certain bacteria.

ENZYMES AND DISEASE

Enzymes are intimately connected with disease. Just what their connection is we do not yet fully understand. Investigation of cancerous tissue has shown that the concentrations of enzymes differ from those in normal tissue but has not shown anything more. When cancer of the prostate occurs with metastases the blood is very high in acid phosphatase, an enzyme which is most active at pH 5.0. Alkaline phosphatase of the blood is found to increase in parathyroidism, various diseases of the bones, and jaundice. Amylase and lipase are increased when there is fibrosis of the pancreas.

Enzymes are somehow concerned with aging and therefore are of interest to the student of geriatrics. The enzymes of bacteria that produce lactic acid are responsible for tooth decay.

In order to treat disease in a purely scientific manner we must first thoroughly understand all physiological phenomena. This requires a knowledge of enzymes and the chemical reactions which they bring about.

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[1] The author has intentionally refrained from inserting the hundreds of references which could have been employed in this article. Readers are referred to "The Enzymes," edited by SUMNER, J. B., AND K. MYRBACK, Academic Press, Inc., New York, **1950**.

STABILIZATION OF OXIDATION STATES THROUGH COORDINATION[1]

BODIE E. DOUGLAS
The Pennsylvania State College, State College, Pennsylvania

THE activity in the field of coordination compounds is very great at the present time as a result of recognition of coordination and coordination compounds as extremely interesting and fertile sources of fundamental knowledge. The significance of coordination compounds in life processes and in the chemistry of practically all metals is now well known. It is generally recognized (although frequently forgotten) that simple ions are incapable of existence except under special circumstances such as in the gaseous state. The life of such gaseous ions is very short. In solution, all ions are combined with the solvent to some extent, so that the chemistry of most metals, as we know it, is the chemistry of the aquated ions. Because the number of water molecules attached to the ions is indefinite or not known, it is common practice to omit the water molecules in writing the formulas of ionic species in solution. The omission of water from the formulas is justifiable only as long as one does not lose sight of the fact that we are not dealing with simple ions.

The term *oxidation state* is used here in preference to the more common term *valence* because it avoids the ambiguity encountered in determining the valence of a metal in a coordination compound. The oxidation state is equal to the number of electrons needed to change the central atom of a coordination compound to the free metal without causing any oxidation or reduction of the ligand atoms or molecules. In certain cases, some doubt concerning the correct oxidation state of the metal remains, because of the uncertainty of the nature of the coordinating agent in the coordination compound. For example, nitric oxide could coordinate as the free molecule, as the positive ion, NO^+, through loss of an electron to the metal atom, or as the negative ion, NO^-, through gain of an electron from an electropositive atom. The evidence is in favor of coordination of the positive ion in most cases through transfer of one electron from the nitric oxide molecule to the central atom. Only in the ion $[Co(NH_3)_5(NO)]^{++}$ (*49*) does there appear to be any very definite evidence for the coordination of the NO^- ion. For a more detailed consideration of inorganic nitrosyl compounds, see ref. (*51*).

The oxidation state of zero for metals in the volatile carbonyls was rather difficult for many chemists to accept because it was so unusual. However, the best evidence is in favor of an oxidation state of zero for these compounds (*7*). The isolation of the compounds $K_4[Ni(CN)_4]$ and $K_4[Pd(CN)_4]$ lends support to the existence of compounds containing metal atoms in an oxidation state of zero (*10, 18*).

The phrase *valence stabilization through coordination* has been widely used (*8, 13*) to designate the same phenomenon discussed here. By stabilization of an oxidation state through coordination, we mean stabilization relative to the aquated ion. If we say that some coordinating agent stabilizes a particular oxidation state of a metal, we really mean that this coordinating agent forms a compound with the metal ion which is more stable toward oxidation or reduction than the aquated ion. If the aquated ion of a particular oxidation state of a metal cannot be isolated, then the mere isolation of a coordination compound containing the metal in the unusual oxidation state is indicative of stabilization of that oxidation state through coordination. As an example, the "simple" or aquated palladium (IV) ion is unknown, but the compound $K_2[PdCl_6]$ has been isolated. In other cases, where the aquated ions are known, the stabilization of an oxidation state can be expressed as a stabilization of that oxidation state to oxidation and/or reduction. If coordination stabilizes a particular oxidation state relative to the aquated ion, then the reduction of the coordination compound to the free metal will be more difficult than the reduction of the aquated ion. Coordination thus results in a shift in the standard potential for this reduction process to a more negative value. The shift of the standard potential is directly related to the logarithm of the dissociation constant of the coordination compound. For the situation in which two oxidation states are involved, the shift of the standard potential for the couple is related to the ratio of the dissociation constants of the coordination compounds of the metal in the two oxidation states. The shift can be in either direction depending on which oxidation state is stabilized to the greater extent.

The stability of a coordination compound as expressed by the dissociation constant is not necessarily directly related to the thermal stability. In general one would expect the ammoniates with small dissociation constants, as found in aqueous solution, to have a greater thermal stability than those which are found to dissociate to a greater extent in aqueous solution. The thermal stability of many coordination compounds containing organic ligands might be limited by the thermal stability of the organic ligand itself. Stability toward chemical attack can be related frequently to the ten-

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TABLE 1
Standard Potentials

Couple	*Complexing agent*	*E°*
Mn(III)-Mn(II)	H_2O	*ca.* 1.51
	CN^-	−0.22
Cr(III)-Cr(II)	H_2O	−0.41
	CN^-	−1.28
	SCN^-	−0.55
Fe(III)-Fe(II)	H_2O	0.77
	o-Phenanthroline	1.14
	CN^-	0.36
Co(III)-Co(II)	H_2O	*ca.* 1.8
	NH_3	+0.1
	CN^-	−0.83
Cu(II)-Cu	H_2O	−0.35
	Ethylenediamine	−0.93
	Diethylenetriamine	−0.97
Cu(II)-Cu(I)	H_2O	−0.17
	CN^-	*ca.* 1.12
Cd(II)-Cd	H_2O	−0.40
	CN^-	−0.90
	NH_3	−0.60
	Ethylenediamine	−0.76
	Diethylenetriamine	−0.82

dency of the complex to dissociate if all other equilibria are also considered (e. g., decomposition of many ammoniates in acid solution and precipitation of metal sulfides from complexes of heavy metals). In those cases where the complexes are not in equilibrium with the aquated metal ions, the dissociation constant has no significance and cannot be evaluated. For example, the hexamminechromium (III) ion is not in equilibrium with the aquated metal ion and slowly decomposes in aqueous solution due to aquation and hydrolysis. For such compounds, one usually bases the estimate of the "stability" of the compound on chemical reactivity, although equilibrium may never be attained. This is somewhat inconsistent, but if we based all our ideas of stability solely on observations under equilibrium conditions, many of our concepts regarding stability of familiar compounds would require revision.

TABLE 2
Electronic Configuration of Some Metal Ions

Metal	*Spatial arrangement*	*3d*	*4s*	*4p*	*5s*
Cr(0)		····0	:	000	
Cr(0)	Octahedral	:::[00]	[0]	[000]	
Cr(III)		···00	0	000	
Cr(III)	Octahedral	···[00]	[0]	[000]	
Mn(0)		·····	:	000	
Mn(I)	Octahedral	:::[00]	[0]	[000]	
Fe(II)		:····	0	000	
Fe(II)	Octahedral	:::[00]	[0]	[000]	
Fe(III)	Octahedral	::·[00]	[0]	[000]	
Co(II)		:::··	0	000	
Co(II)	Octahedral	:::[00]	[0]	[000]	
Co(III)	Octahedral	:::[00]	[0]	[000]	
Ni(0)		:::··	:	000	
Ni(0)	Tetrahedral	:::::	[0]	[000]	
Ni(II)	Tetrahedral	:::··	[0]	[000]	
Ni(II)	Planar	::::[0]	[0]	[00]0	
Cu(I)	Tetrahedral	:::::	[0]	[000]	
Cu(II)		:::··	0	000	
Cu(II)	Planar	::::[0]	[0]	[00]·	

The dissociation constant of a coordination compound can be obtained by calculation from polarographic data (*36*) or from other electrochemical data. Dissociation constants can also be evaluated from solubility data or by the measurement of physical properties such as light absorption which enable one to evaluate the concentration of the species present in solution. Bjerrum (*6*) has introduced a valuable method for the determination of step-wise formation constants which is based on pH measurements.

The use of coordination as a means of stabilization of oxidation states is an especially useful method for effecting the stabilization of unusual oxidation states. No other method is of such general applicability. The various methods which are available for the stabilization of oxidation states have been reviewed by Kleinberg (*32, 33*).

CONTRIBUTING FACTORS

The factors contributing to stability are numerous and it is difficult to evaluate the exact contribution of each. Many of the factors are interrelated and cannot be considered completely independent of one another. However, these factors will be considered separately in order to obtain some concept of the extent to which variation of each of these factors affects stability.

Electronic Configuration

Availability of Orbitals for Bond Formation. The electronic configuration of the central ion seems to be one of the most important factors in determining the type and stability of coordination compounds formed. The necessary orbitals must be available for the formation of bonds of sufficient strength and number to give the resulting compound the maximum stability. (See Table 5 for a summary of some hybridized bonding orbitals and Table 2 for the electronic configuration of some metal ions). Coordination compounds are to be expected less frequently when the most stable orbitals are not available for bond formation. It is possible, however, to vacate orbitals for bond formation by the promotion of unpaired electrons to a higher level. It is also possible to form compounds involving ionic bonds or compounds of lower coordination number, thus it would be expected that compounds of manganese (0) with a coordination number of six would be unstable since this would require the pairing of two *s* electrons with *d* electrons and the promotion of one *d* electron to a higher level or the promotion of the two *s* electrons which is improbable. A lower coordination number is possible, but a closed electronic configuration could never be achieved. Similarly,

compounds of iron (I) with a coordination number of six should be unstable and only the unstable $M_3[Fe(CN)_5NO]$ (M is a univalent metal) which presumably contains iron (I) has been reported (*45*, *46*). An iron (0) compound with a coordination number of five or less, such as $Fe(CO)_5$, should be expected. The cobalt (II) compounds with a coordination number of six are quite unstable with respect to oxidation to cobalt (III). This is to be expected since the donated electrons would be required to occupy at least one *4d* orbital or the odd electron in the *3d* orbital must be promoted to the less stable *4d* orbital. Either possibility leads to instability. Cobalt (II) can accommodate electrons to form square coplanar or tetrahedral complexes. The cobalt (−1) ion should be able to accommodate electrons occupying the sp^3 orbitals to form tetrahedral complexes.

Nickel (II) would require the promotion of two *3d* electrons to some higher level for the formation of six covalent bonds. One would expect these two unpaired electrons to be easily lost to form nickel (IV), but this is not true and apparently the bonds in such compounds are predominately ionic in character. Nickel (II) can still form compounds with a coordination number of four which have a planar or tetrahedral configuration. Pauling (*55*) has shown that the planar configuration gives greater stability for nickel (II). Orbitals are available for the formation of sp^3 bonds in the nickel (I) and nickel (0) compounds. Palladium (II) and platinum (II) compounds having a planar configuration are known to be very stable. Palladium shows the tendency to lose two electrons to form an octahedral compound only in the case of $M_2[PdCl_6]$. This tendency is very pronounced in the case of platinum.

Compounds of copper, silver, and gold in oxidation states below +3 would be expected to be tetrahedral for a coordination number of four unless one *d* electron is promoted to a higher level. Square coplanar complexes are known for silver (II) and copper (II) (*48*).

Some of the above observations regarding the stable electronic configurations of coordination compounds have been restated by Pauling in a slightly different way. In the same paper (*55*) he also introduced some new ideas about bond formation in coordination compounds. Pauling states that a nonbonding electron occupying an orbital may be moved into a less stable orbital without a net loss in energy of the system, in case that the orbital that it vacates is used for formation of a good covalent bond, whereas an unshared pair occupying a stable orbital cannot be easily moved into a less stable orbital.

Of greater significance is the suggestion that stable compounds can be formed even though the central atom has fewer vacant orbitals than the number of ligands coordinated. The maximum stability for a complex in which a central atom forms several partially covalent bonds with surrounding atoms is obtained when the central atom has available a bond orbital for each of the ligands; but in case the number of bond orbitals is less than the number of ligands somewhat less stable structures can also result from the resonance of the bond orbitals among the ligands. The maximum stability for a hexacoordinate complex ion is achieved when there are six orbitals to resonate among the six ligands. Fewer resonance structures result when there are fewer orbitals available and the resulting complex ion is less stable.

Thus nickel (II) could form partially covalent bonds using *4s* and *4p* orbitals in its hexacoordinate compounds without promoting its two unpaired *d* electrons to higher orbitals. This also offers a possible explanation for the partial covalent character of the compounds of zinc and cadmium showing a coordination number of six, even though the *d* orbitals of lower quantum number are completely filled.

The sharing of a limited number of orbitals among a greater number of ligands permits the continuous transition from ionic bonding to a fairly definite limit of covalency where all of the available orbitals are occupied (shared) by all of the ligands. The magnetic criterion of bond type gives no information about the maximum covalent contribution for limiting ionic structures when the ionic and purely covalent structures differ in the number of unpaired electrons. Nor can one determine the maximum ionic contribution for limiting covalent structures in similar cases. Of course, the magnetic method cannot be used even to distinguish between the limiting structures if they do not differ in the number of unpaired electrons. We only know that the transition from the extreme ionic structure to the extreme covalent structure must be discontinuous in those cases where there is a difference in the number of unpaired electrons in the two structures. It might be possible to determine an estimate for the maximum covalency to be expected in "ionic" compounds from the number of orbitals which can be shared among the ligands.

The last rule which Pauling gives is that there is a special stability associated with having no electrons, half its complement of electrons, or a full complement of electrons in a subgroup of orbitals; and this subgroup may be an atomic subgroup of *p*, *d*, or *f* orbitals, or the residuum of the subgroup after some of its orbitals have been used for bond formation. As examples, chromium (III) has three electrons for the three *d* orbitals not required for bond formation, whereas cobalt (III) has six electrons for the three orbitals. The situation where three *d* orbitals are empty in a complex ion is not normally encountered, unless the six-coordinate cadmium and zinc compounds utilize the *d* orbitals of the same quantum number as the *s* and *p* orbitals used for bond formation.

Effective Atomic Number. The concept of "effective atomic number" (e. a. n.) was introduced by Sidgwick to denote the total number of electrons, both shared and unshared, surrounding an atom. It has been assumed that chemical combination for any element can be explained as an approach to the effective atomic number

of an inert gas. The tendency to attain the effective atomic number of an inert gas is apparent in many simple compounds, but breaks down for simple compounds of the elements in the middle groups of the periodic table. Coordination compounds of many of the elements in the middle groups of the periodic table conform to this concept, but even here there are many exceptions.

The coordination compounds of many of the group VIII metals show a pronounced tendency of the metals to attain the effective atomic number of an inert gas.

TABLE 3

Complex ion	*Oxidation state*	*E. a. n.*	*Complex ion*	*Oxidation state*	*E. a. n.*
$M_3[Fe(CN)_6]$	+3	35	$M_3[Co(CN)_6]$	+3	36
$M_4[Fe(CN)_6]$	+2	36	M [$Co(CO)_4$]	−1	36
$M_3[Fe(CN)_5CO]$	+2	36	[$Co(CO)_3NO$]	−1	36
$M_3[Fe(CN)_5NO]$	+1	37	$M_3[Rh(CN)_6]$	+3	54
[$Fe(CO)_5$]	0	36	$M_3[Ir(CN)_6]$	+3	86
$M_2[Fe(CO)_4]$	−2	36	$M_2[Ni(CN)_4]$	+2	34
$M_4[Ru(CN)_6]$	+2	54	$M_3[Ni(CN)_3]$	+1	33
$M_2[Ru(CN)_5NO]$	+2	54	$M_4[Ni(CN)_4]$	0	36
$M_2[RuCl_5NO]$	+2	54	[$Ni(CO)_4$]	0	36
[$Ru(CO)_5$]	0	54	$M_2[PdCl_6]$	+4	54
$M_2[OsCl_6]$	+4	84	$M_2[Pd(CN)_4]$	+2	52
$M_3[OsCl_6]$	+3	85	$M_4[Pd(CN)_4]$	0	54
$M_2[OsCl_5NO]$	+2	86	$M_2[PtCl_6]$	+4	86
$M_4[Os(CN)_6]$	+2	86	$M_2[Pt(CN)_4]$	+2	84
$M_4[Co(CN)_6]$	+2	37			

From Table 3 it is apparent that the iron (II) and iron (0) compounds have the effective atomic number of krypton. The iron (III) compounds give iron an e. a. n. of 25 and are, in a number of cases, somewhat less stable than the corresponding iron (II) compounds. However, iron (III) is stabilized to a greater extent than iron (II) by coordination with cyanide ion and with those groups which tend to form ionic bonds with iron. The unusual compound shown in which iron has an oxidation state of +1 is much less stable (*43*, *46*) than the corresponding iron (II) compound. In ruthenium (II), ruthenium (0) and osmium (II) compounds the metals likewise have the e. a. n. of inert gases. As in the case with iron, α,α'-dipyridyl stabilizes the divalent state of osmium and ruthenium. The compound $[Ru(dipy)_3]Cl_2 \cdot 6H_2O$ is remarkably stable; it can be boiled with 50 per cent caustic alkali without decomposition (*11*). The e. a. n. of ruthenium and osmium compounds in higher oxidation states fall short of the values for inert gases. However, it does not seem unlikely that compounds of ruthenium (VI) and osmium (VI) with a coordination number of eight would be capable of existence. There are two electrons in the outer d orbitals in each case so that four d orbitals, the s and three p orbitals would be available for the formation of d^4sp^3 bonds. The stability of such a compound should be enhanced by the high bond strength and the closed electronic configuration of the central ion.

Simple cobalt (III) compounds are very unstable, but coordination results in such a drastic change in the standard potential for the cobalt (II)-cobalt (III) couple that the coordination compounds of cobalt (III) are the more stable of the two oxidation states. Cobalt (III) compounds with a coordination number of six have an effective atomic number of 36 as do the cobalt carbonyl hydride (*62*) and cobalt nitrosyl carbonyl. The cobalt in the latter two compounds has an apparent oxidation state of −1 (*7*, *51*). The very stable rhodium (III) and iridium (III) compounds with a coordination number of six also have the e. a. n. of inert gases.

The compounds of nickel (II) have an e. a. n. of 34 or 38, but never 36. However, the nickel (0) compounds as exhibited by nickel carbonyl and the unusual $K_4[Ni(CN)_4]$ give nickel an e. a. n. of 36. Palladium (II) and platinum (II) likewise do not have the e. a. n. of inert gases, but this is achieved in the palladium (0), palladium (IV), and platinum (IV) compounds.

The only coordination compound of manganese in which manganese has an e. a. n. of 36 is the compound $K_5[Mn(CN)_6]$, which is obtained by reducing the corresponding manganese (II) compound with aluminum (*46*). The manganese (I) compound is not particularly stable, but it is remarkable that it exists at all. Rhenium forms a carbonyl chloride, $[Re(CO)_5Cl]$ (*63*), and a dimeric carbonyl, $[Re(CO)_5]_2$ (*26*) in which the e. a. n. of rhenium is 86.

Chromium, molybdenum, and tungsten form hexacarbonyls in which each metal has the effective atomic number of an inert gas. In no other coordination compound does chromium attain the e. a. n. of 36, but it seems likely that a chromium (0) cyanide complex similar to those of nickel and palladium might be prepared. The d^2sp^3 orbitals would be available for bond formation, giving the chromium the same electronic configuration as that found in the hexacarbonyl. The attempted reduction of $K_3[Cr(CN)_6]$ with metallic potassium in liquid ammonia was reported to yield indefinite products (*10*). Molybdenum (IV) and tungsten (IV) form octacyanides in which the e. a. n. of the metal in each case is that of an inert gas.

The metals in the compounds of copper (I), silver (I), zinc (II), and cadmium (II) with a coordination number of four have the e. a. n. of inert gases.

From the compounds mentioned here, it is evident that the attainment of the e. a. n. of an inert gas is frequently associated with the stability of coordination compounds and this concept is not without value. It is important to note that most of the compounds cited here are cyanides, carbonyls, and partially substituted nitrosyls. In some cases the cyanides are merely representative of a wide variety of coordination compounds, e. g. cobalt (III), while for some other elements, cyanide ion is the only group known to give a compound of the type shown, e. g. manganese (I). Cyanide complexes are useful for this sort of comparison because cyanide ion is one of the best coordinating agents known for such a wide range of metals (see Table 4). The carbonyls and the nitrosyl substituted

compounds are not representative of a group of compounds for any one element. These latter compounds conform to the e. a. n. concept better than any other class of coordination compounds.

TABLE 4
Representative Compounds

Metal	*Cyanide*	*Amine*	*Carbonyl*	*Halide*
Cr(0)	...	...	$Cr(CO)_6$	...
Cr(III)	$M_3Cr(CN)_6$	$Cr(NH_3)_6Cl_3$	...	...
Mn(I)	$M_5Mn(CN)_6$	...	...	...
Mn(I)	$M_2Mn(CN)_5$	...	...	...
Mn(II)	$M_4Mn(CN)_6$	...	...	$MMnX_3$
Mn(III)	$M_3Mn(CN)_6$	...	...	M_2MnX_5
Fe(−2)	...	...	$M_2Fe(CO)_5$	...
Fe(0)	...	...	$Fe(CO)_5$	...
Fe(II)	$M_4Fe(CN)_6$	$Fe(o\text{-phen}^a)_3Cl_2$	...	$MFeCl_3$
Fe(III)	$M_3Fe(CN)_6$	$Fe(o\text{-phen})_3Cl_3$	...	M_2FeCl_5
Ru(0)	...	...	$Ru(CO)_5$	...
Ru(II)	$M_4Ru(CN)_6$	$Ru(dipy^b)_3Cl_2$	...	...
Os(II)	$M_4Os(CN)_6$	$Os(dipy)_3Cl_2$	...	...
Os(III)	...	...	...	M_2OsCl_5
Co(−1)	...	...	$MCo(CO)_4$	...
Co(II)	$M_4Co(CN)_6$	$Co(NH_3)_6Cl_2$	...	M_2CoCl_4
Co(III)	$M_3Co(CN)_6$	$Co(NH_3)_6Cl_3$	...	...
Rh(III)	$M_3Rh(CN)_6$	$Rh(NH_3)_6Cl_3$	...	M_3RhCl_6
Ir(III)	$M_3Ir(CN)_6$	$Ir(NH_3)_6Cl_3$	...	M_3IrCl_6
Ni(0)	$M_4Ni(CN)_4$	...	$Ni(CO)_4$	...
Ni(I)	$M_2Ni(CN)_2$	...	...	...
Ni(II)	$M_2Ni(CN)_4$	$Ni(NH_3)_4Cl_2$	...	...
Pd(0)	$M_4Pd(CN)_4$	...	...	...
Pd(II)	$M_2Pd(CN)_4$	$Pd(NH_3)_4Cl_2$	...	M_2PdCl_4
Pd(IV)	...	...	...	M_2PdCl_6
Pt(II)	$M_2Pt(CN)_4$	$Pt(NH_3)_4Cl_2$	...	M_2PtCl_4
Pt(IV)	...	$Pt(NH_3)_6Cl_4$	...	M_2PtCl_6

[a] *o*-phenanthroline.
[b] dipyridyl

Although the e. a. n. concept is useful, it is obvious that other factors must be even more important in determining stability. The most stable coordination compounds of nickel, palladium, copper, manganese, chromium, and many other metals are not those for which the e. a. n. of the central ion is that of an inert gas. On the basis of the e. a. n. concept one would predict compounds such as $[Ni(NH_3)_6]^{++}$ which are not encountered. The availability of the proper electron orbitals for the formation of covalent bonds of the greatest strength is more important than the attainment of the e. a. n. of an inert gas. Those compounds which conform to the e. a. n. concept are almost always those in which the metal ion has available the necessary electron orbitals for the formation of favorable hybridized bonds.

Stable compounds are also found in which the metals have too few electrons to attain the e. a. n. concept of an inert gas. Thus, chromium (III), ruthenium (III), osmium (III), nickel (II), palladium (II), and copper (II) are the stable oxidation states for these metals and their compounds do not conform to the e. a. n. concept. Certain groups such as cyanide ion stabilize iron (III) to a greater extent than iron (II) (as compared with the aquo complexes) although the iron (II) compound conforms to the e. a. n. concept.

OXIDATION STATE OF CENTRAL ATOM

It is difficult to correlate the effect of the oxidation state of metals with the stability of coordination compounds. A system which holds for one metal might fall down completely when applied to another metal. The oxidation state of the metal is probably most important in so far as it affects ion size, the electronic configuration, and the orbitals available for bond formation. These factors are discussed elsewhere.

Oxidation states far removed from the normal oxidation state are not often encountered because of their strong oxidizing or reducing properties, *e. g.* $[Mn(CN)_6]^{-5}$, $[Ni(CN)_4]^{-4}$. Oxidation states which would normally be considered very high for an element usually are encountered only with very strong coordination agents, *e. g.* $[Ag(o\text{-phen})_2]^{++}$ or with a strong coordinating agent such as the oxide ion which itself is oxidized only with great difficulty, *e. g.*, MnO_4^-, $CrO_4^=$, and $FeO_4^=$.

Pauling (*56*) states that in a complex ion all atoms except hydrogen and the most electropositive and the most electronegative tend to form bonds of such number and amount of ionic character as to make its residual charge very close to zero. On the basis of the difference in electronegativities, the bond between oxygen and a transition metal would have about $^1/_3$ covalent character. For the hexaquo ions containing six metal-oxygen bonds, the residual charge on the central atom would be zero only for an oxidation state of two for the metal. An oxidation state of two is found to be the most common for five of the seven metals from chromium to zinc, namely, Mn, Co, Ni, Cu, and Zn. On this basis, an increase in covalent character of the metal-ligand bond would favor a higher oxidation state for the central atom. There is some evidence in this direction but it is not striking. Thus, coordination with cyanide ion stabilizes the oxidation state of three for iron, cobalt, and manganese. However, amines in general do not stabilize an oxidation state higher than the normal oxidation state for the aquated ion of these metals except in the case of cobalt where factors such as electronic configuration are probably of greater importance.

Sidgwick (*65*) states that, "an increase of valency increases the tendency to covalency formation, while on the other hand a high valency involves a still higher covalency in the complex, so that it restricts both the number and (since the highest covalencies are less stable) the stability of the complexes formed."

BOND TYPE

Bond type is an important factor in determining the stability of coordination compounds. Coordination compounds containing covalent bonds usually give the greatest preferential stabilizing effect to a particular oxidation state of a metal. Since an increase in charge and a decrease in size of the central

ion favor the formation of covalent bonds, it would be expected that higher oxidation states would be favored in covalent coordination compounds. However, an increase in oxidation state increases the normal coordination number for the central ion and with the decrease in size this can be a limitation. The tendency to form the most favorable bonds and achieve a stable electronic configuration are probably much more important than polarization effects. For cobalt, the factors contributing to stability work together since the cobalt (III) ion is not only smaller and bears a higher charge than the cobalt (II) ion, but a completed electronic configuration also results. In the case of iron, coordination with cyanide ion stabilizes iron (III) with respect to iron (II) although the iron (II) complex has the completed electronic configuration. These cases are in line with Fajan's views and with Pauling's principle of electroneutrality, since a higher oxidation state would be necessary to neutralize the greater negative charge transferred to the metal atom as a result of the increase in covalent character of the bonds. In the case of the cyanides, Pauling also emphasizes the importance of double bond formation between the metal ion and carbon in reducing the residual negative charge on the metal.

The situation in which lower oxidation states are preferentially stabilized are the exceptions to the rules mentioned. In these cases the electronic configuration and in certain instances the ion size must be particularly important. The stabilization of iron (II), osmium (II), and ruthenium (II) by coordination with *o*-phenanthroline and α,α'-dipyridyl are the most noteworthy examples. Here the completed electronic structures probably contribute very greatly to the stability.

The formation of coordination compounds involving ionic bonds will generally, if not always, stabilize the higher of two relatively stable oxidation states. This is to be expected since small size and high charge of the central ion are the major factors contributing to electrostatic attraction and hence to the stability of ionic complexes. Specific or selective behavior of coordinating agents with certain metals or certain oxidation states (other than the stabilization of the higher of two moderately stable oxidation states) is not to be expected if ionic bonds are involved in the resulting compound.

According to Fajans (*19*) an electrovalence tends to become a covalence due to large polarization forces when the following conditions are satisfied: (1) The cation is small or the anion is large, (2) The charge on either ion is large, and (3) The polarization will be less for a cation having an inert gas electronic configuration than for cations with other electronic configurations.

However, it is misleading to assume an electrostatic type of binding and apply these rules directly to the situation encountered in many coordination compounds. On the basis of electrostatic forces, beryllium and aluminum, because of their small size, would be expected to be the divalent and trivalent ions forming the most stable coordination compounds. However, this is known to be false. It is also difficult to imagine how polarization forces alone could distort a normal tetrahedral arrangement of electrostatic bonds into a square coplanar configuration such as is found for many nickel, palladium, and platinum compounds. Nor does it seem likely that polarization forces could result in a planar configuration for compounds of nickel (II) and copper (II) but not in the case of zinc (II), manganese (II), etc. These ions are similar in size and charge and none has an inert gas configuration.

Many coordination compounds containing cyanide ion or *o*-phenanthroline offer good examples of covalent type bonding and great stability. The coordination compounds with fluoride ion are usually of the ionic type and stabilize the higher of the common oxidation states among the metals. The hydroxide and oxide ions are among the best coordination agents for stabilization of very high oxidation states. This is due to the difficulty with which the hydroxide and oxide ions are oxidized and the increased acidity of metals in very high oxidation states as a result of the decreased size and increased charge of the ions. The oxide ion is a better coordination agent than the hydroxide ion and the hydroxy-compounds formed in basic solution show a great tendency to form oxycompounds due to the increased acidity of the hydroxide ion after coordination. There is a universal decrease in the stability of oxy-anions of metals in acid solution, as one would predict on the basis of the decrease in the concentration of the coordination agent.

The differences in the electronegativity of the central atom and the coordinated atom has considerable bearing on the bond type of coordination compounds. Ionic bonds result from the coordination of atoms of widely different electronegativities. It is apparent that covalent bonds cannot be formed unless the necessary orbitals are available for the shared electron pairs or can be made available without the expenditure of a great deal of energy.

Copley, Foster, and Bailar (*13*) point out that all good oxidation-reduction indicators for the titration of ferrous ion are examples in which stabilization of low oxidation states is achieved. This is to be expected from a consideration of bond type. Among the requirements of a good oxidation-reduction indicator are great stability and intense coloration. Both requirements are best fulfilled by covalent coordination compounds and preferential coordination of a lower oxidation state can occur only in the case of covalent compounds.

Whenever resonance is possible in a coordination compound, greater stability results. Larger organic molecules with conjugated double bonds such as *o*-phenanthroline and α,α'-dipyridyl are excellent examples of this stabilization. Although the coordination compounds of iron with most amines are not particularly stable, the iron (II) compounds with *o*-phenanthroline and α,α'-dipyridyl are amazingly stable. It has been mentioned that these organic molecules also stabilize ruthenium (II) and osmium (II)

to a marked extent. It is interesting to note that in these coordination compounds the metal ions have closed electronic configurations. Very stable coordination compounds of *o*-phenanthroline and α,α'-dipyridyl have been obtained for a number of other metals. Some importance has been attached to the apparent selectivity of these organic molecules for divalent ions. It is true that stabilization is very marked in the case of iron (II) and the normally unstable silver (II). However it is probably more important that strong bonds are formed to stabilize the particular oxidation state as a result of a stable electronic configuration and suitable ionic radius for the fairly rigid chelating agents.

A recent attempt to prepare a gold (II) compound with *o*-phenanthroline or α,α'-dipyridyl by Block (*9*) was unsuccessful. Cobalt (II) is not stabilized by *o*-phenanthroline in preference to cobalt (III) (*53*). Unpublished work of the author (*16*) indicates that *o*-phenanthroline and α,α'-dipyridyl stabilize chromium (III) to a greater extent than chromium (II) contrary to the report in the literature (*24*).

Compounds containing groups such as cyanide ion, for which it is possible to write double bonds between the metal and carbon atom, usually lead to great stability. The resonance involving double bonds probably enhances the stability of the compounds.

Stereochemistry

Stable Configurations. The importance of the formation of bonds of great stability has already been mentioned. However, the relationship of the bond strength to the resulting configuration has not been emphasized. The distribution in space of the various types of bonding orbitals and combination of orbitals (hybridized orbitals) (*55*) have been summarized by Kimball (*31*) and the relative strength of the resulting bonds have been presented by Pauling (*55*) as a result of quantum mechanical considerations (Table 5).

TABLE 5
Some Hybridized Bonding Orbitals

Orbitals	*No. of bonds in set*	*Distribution in space*	*Bond strength*
s	1	Nondirectional	1
sp or dp	2	Linear	...
p^3	3	Trigonal pyramid	1.732
sp^2, d^2s, dp^2, or d^3	3	Trigonal plane	...
sp^3	4	Tetrahedron	2
d^3s	4	Tetrahedron	2.236
dsp^2	4	Square coplanar	2.694
d^2sp^3	6	Octahedron	2.923
sdp	3	Unsymmetrical plane	3
d^3fsp^3	8	Cube	...
d^3f^4s	8	Cube	...
d^4sp^3	8	Dodecahedron	...
d^4p^3	8	Antiprism	...
d^4sp^2	8	Face-centered prism	...

There is always a tendency for a metal ion to acquire the electrons necessary to fill its vacant orbitals and also to form bonds of maximum strength. In many cases both factors work hand in hand and the compounds formed are very stable. However, there are still exceptions such as the stabilization of iron (III) over iron (II) by coordination with cyanide ion, although the iron (II) compound gives iron a closed electronic configuration. The high bond strength of configurations such as result from hybridization of dsp^2 orbitals is probably a major factor in contributing to the stability of coordination compounds such as the covalent compounds of nickel (II) which do not have completely filled electron shells.

TABLE 6

Type compd.	*Coordination number*	*Configuration*	*Radius ratio*
AB_2	2	Linear	$A/B > 0$
AB_3	3	Triangular	$A/B > 0.15$
AB_4	4	Planar	$A/B > 0.41$
AB_4	4	Tetrahedral	$A/B > 0.22$
AB_6	6	Octahedral	$A/B > 0.41$
AB_8	8	Antiprismatic	$A/B > 0.65$

Coordination Number. There is a limit to the number of groups which can be coordinated around a central ion. These limitations are due to the relative sizes of the atoms involved and the electronic orbitals available. Sidgwick has proposed a set of rules which set forth the limits of maximum covalence (*64*). Thus the maximum covalence of hydrogen is two. The elements in the first short period (Li–F) do not have *d* orbitals available and the maximum covalence is four. The elements in the second short period (Na–Cl) and first long period (K–Br) have *d* orbitals available as well as *s* and *p* orbitals; the maximum covalence is six. For the remaining periods, the maximum covalence is eight.

It is obvious that the maximum coordination number will not always be attained if the coordinating groups are so large or the central atom so small that the maximum number of groups cannot be packed around the central atom without loss of contact between the bonded atoms. Since these limits are strictly geometrical the limiting ratios of the sizes of the central atom to the coordinated atom can be calculated for each configuration. The radius ratio rules (*20*) are given in Table 6. Many elements exhibit their maximum coordination number only with very small coordinating groups such as the fluoride ion (*e. g.*, SiF_6^{--} and TaF_8^{-3}).

Compounds in which the central ion achieves its maximum coordination number as given by the radius ratio rules are usually more stable than those in which fewer groups are coordinated. However, the coordination number is probably less important in determining the stability of coordination compounds than other factors as long as strong bonds are formed and the central ion is completely surrounded.

Chelation. Coordinating groups which contain two ligand atoms are called chelate groups. The formation of chelate rings enhances the stability of the resulting compound. For instance, ammonia forms many stable coordination compounds with several different ele-

ments, but the alkyl amines are very poor coordinating agents. However, an aliphatic chain containing two amine groups (*e. g.*, ethylenediamine) coordinates with many metals to form compounds considerably more stable than those containing ammonia. In general, five- and six-membered chelate rings are more stable than the larger and smaller rings. Since the angle subtended at the metal ion is 90° for a regular octahedron or a square coplanar structure, the remaining angles can be calculated for rings of varying size. A four-membered ring results in angles of 90°, a five-membered ring has four angles of about 112° each and the six-membered ring has five angles which are somewhat larger than 112° each, allowing for puckering of the ring. The 112° angle for the five-membered ring is nearest the tetrahedral angle 109° and this ring size would be expected to be most favorable for most organic molecules. The five-membered rings are usually the most stable. Most of the only well-established coordination compounds containing stable four-membered chelate rings are represented by the carbonato compounds. Rings containing more than six members are rarely encountered, possibly because they are somewhat less probable and have not been sought after by most investigators.

Certain chelate groups with rigid structures which conform to the stable configuration of the metal ions give especially stable compounds. The correct spatial arrangement and rigidity of the coplanar *o*-phenanthroline molecule doubtless contributes to the stability of its compounds. The phthalocyanine molecule has also been shown to be coplanar. There is just room in the center of the molecule for a metal atom such as copper (II) which is bonded to four nitrogens at the corners of a square. The phthalocyanine derivatives of metals are remarkably stable. The copper derivative can be sublimed unchanged at 550° (*15*). In addition to the contribution of chelation to the stability of the compound, the complete cycle of conjugated double bonds must also stabilize the molecule as a result of resonance. Several metals which do not normally form square coordination compounds form quite stable coplanar compounds with phthalocyanine. Because of the limited space available for the metal ion in the phthalocyanine molecule, there is a possibility of stabilizing some oxidation state higher than two for a metal which is too large in the divalent state.

Table 7 gives the relative stabilities of some coordination compounds of copper and cadmium to illustrate the effect of chelation (*17, 37*).

Steric Factors and Forced Configurations. It has been pointed out that large groups may prevent an ion from exhibiting its maximum coordination number. Steric factors can also prevent a metal ion from assuming the most desirable distribution of bonds. The phthalocyanine molecule forces several metals which normally give tetrahedral complexes into a planar configuration. The quadridentate amine, tris(2-aminoethyl)amine, forces the distribution of bonds into an irregular tetrahedral arrangement. Compounds have been formed

TABLE 7

Complex	K_d (*Dissociation constant of complex*)
$Cd(NH_3)_4^{++}$	3.3×10^{-7}
$Cd(en^a)_3^{++}$	6.7×10^{-13}
$Cd(pn^b)_3^{++}$	5.4×10^{-13}
$Cd(dien^c)_2^{++}$	7.6×10^{-15}
$Cd(py^d)_4^{++}$	3.2×10^{-3}
$Cu(NH_3)_4^{++}$	4.7×10^{-14}
$Cu(en)_2^{++}$	1.9×10^{-20}
$Cu(pn)_2^{++}$	6.8×10^{-21}
$Cu(dien)_2^{++}$	1.4×10^{-21}

[a] Ethylenediamine. [b] Propylenediamine. [c] Diethylenetriamine. [d] Pyridine.

with this compound and nickel (II), palladium (II), and platinum (II) (*47*) as well as other metals. Similarly, interference of methyl groups in the coordination compounds of metals with two molecules of 3,3′-5,5′-tetramethyl-4′4′-dicarbethoxy-dipyrromethene apparently prevents the planar distribution of bonds (*50*). The resulting compound with nickel is paramagnetic which indicates a tetrahedral configuration.

The effect of forced configurations on bond type and stability have not been definitely established. However, an interesting situation is found in the compounds of nickel (II) with stilbenediamine (1,2-diphenylethylene diamine). Both the racemic and optically active stilbenediamine show a greater tendency to form coordination compounds than is shown by the meso base. The coordination compounds containing the meso base are generally poorly crystalline and unstable while those of the racemic and active base are easily prepared and stable. The racemic and active forms of the base tend to form blue paramagnetic complexes with nickel (II), while the meso form tends to give a yellow diamagnetic complex. The yellow diamagnetic complexes are undoubtedly planar, whereas the blue paramagnetic complexes might be tetrahedral or octahedral. The meso base possibly causes more steric hinderance due to the fact that the phenyl groups are on the same side and this results in a decrease in stability. If two water molecules or anions are coordinated to form an octahedral complex, the interference of the phenyl groups might be lessened. The situation is complicated by the fact that a wide variety of compounds were obtained, some similar in composition but differing in color and magnetic behavior (*39, 40*).

Symmetry. Symmetry doubtless contributes to the stability of coordination compounds. Jaeger (*29*) attributes the greater stability of coordination compounds containing optically active coordinating groups of only one configuration to the greater symmetry of the molecule. If one treats the ion $Co(l\text{-cptdn})_2Cl_2^+$ with *d*-cyclopentanediamine (*d*-cptdn) the expected compound containing both *d*- and *l*-cyclopentanediamine is not obtained, but instead a mixture of 2 $Co(l\text{-cptdn})_3^{+++}$ and $Co(d\text{-cptdn})_3^{+++}$ results (*29*). Jaeger is of the opinion that the compound containing the two configurations of an opti-

cally active coordinating group is not stable relative to the other forms because of the low symmetry. Bailar (*2*) has recently reviewed some aspects of this field. The greater symmetry of *trans* isomers is frequently cited as a reason for their greater stability, but the most obvious examples are those in which negative ions are involved and the electrostatic repulsion is probably more important than the symmetry.

Insolubility

Insolubility certainly enhances the stability of any compound and in the case of some coordination compounds it is probably the major factor contributing to stability. The stability of coordination compounds of chromium (II) with hydrazine is probably largely due to their insolubility.

EXAMPLES

The transition metals form the most stable coordination compounds and the transition metals also show the greatest variation in oxidation states. It is of interest to consider some of the outstanding examples of stabilization of oxidation states through coordination. Many of the examples have been mentioned before but are included here in the periodic order of the metals.

Inner Transition Elements

The lanthanides form coordination compounds with several oxygen-containing chelate groups such as acetylacetone, but the stabilization of particular oxidation states is not particularly striking. Among the actinides, the chemistry of uranium is interesting because of the variations of oxidation states. Uranium (III) is characterized by compounds such as $H_3[U(SO_4)_3]$. The oxidation state of four is stabilized by compounds such as KUF_5, $K_3[UX_8]$, U(acetylacetonate)$_4$ (*5*), and $K_4[U(C_2O_4)_4]$ (*35*).

Group VB

Vanadium (II) salts are strongly reducing, but compounds such as $K_4[V(CN)_6]\cdot 3H_2O$ have been characterized (*57*) although they are still strong reducing agents. The higher oxidation states of vanadium are stabilized in the familiar oxy-anions.

Group VIB

The stabilization of chromium (II) in insoluble compounds with hydrazine has already been mentioned.

Tungsten (III) is stabilized by coordination with chloride ion in the compound $K_3[W_2Cl_9]$ (*54*). The oxidation state of four for molybdenum and tungsten is stabilized in the compounds $K_4[Mo(CN)_8]$ and $K_4[W(CN)_8]$ (*12*). The hexacarbonyls of chromium, molybdenum, and tungsten are examples of stabilization of the metals in an oxidation state of zero.

Group VIIB

Cyanide ion forms coordination compounds with manganese (I), (II), and (III). The mere existence of a compound of manganese (I) is evidence of a very great stabilization of this unusual oxidation state (*11*). The existence of an anion with five minus charges is further evidence of an unusual stability associated with the compound $K_6[Mn(CN)_6]$. The compound $K_3[Mn(CN)_6]$ is easily obtained by the air oxidation of the corresponding manganese (II) ion; the shift in the standard potential is of the order of two volts (*16*).

Group VIII

Iron exhibits the unusual oxidation state of plus six in the compound $K_2[FeO_4]$ which can be prepared by oxidation with chlorine in concentrated alkaline solution (*21*, *61*) or by anodic oxidation in alkaline solution (*22*, *23*, *58*, *59*). The oxidation state of zero is exhibited by iron in the pentacarbonyl, while the formal oxidation state of iron in the compound $K_2[Fe(CO)_4]$ has been given as -2. Ruthenium also forms a pentacarbonyl, representing an oxidation state of zero.

The oxidation state of three is very uncommon in simple salts of cobalt, but in coordination compounds the oxidation state of three is commonly encountered. The compounds $K[Co(CO)_4]$ and $Co(CO)_3NO$ have been cited as examples of cobalt in an oxidation state of minus one (*7*, *51*). Cobalt (IV) is found in the heteropolymolybdate $3K_2O\cdot CoO_2\cdot 9MoO_3\cdot 6^1/_2H_2O$ and in some of the binuclear complexes containing peroxide ion as one of the two bridging groups. These latter compounds contain one cobalt (III) and one cobalt (IV) ion (*42*).

Nickel shows oxidation states from zero to three in coordination compounds. The compounds $Ni(CO)_4$ and $K_4[Ni(CN)_4]$ contain nickel (0). Coordination with cyanide ion also stabilizes nickel (I) as shown by the compounds $K_2Ni(CN)_3$ (*4*) and $K_2[Ni(CN)_3CO]$ (*44*). Many nickel (II) compounds are known, but nickel (III) compounds are rare. The compound $NiBr_3(Et_3P)_2$ has recently been reported to be formed by oxidation of the corresponding nickel (II) compound with bromine (*30*). The molecular weight measured in benzene indicated that the molecule is monomeric. Magnetic susceptibility measurements indicate one unpaired electron as would be expected for the nickel (III) ion. The heteropolymolybdate of nickel (IV), $3BaO\cdot NiO_2\cdot 9MoO_3\cdot 12H_2O$, has been characterized (*24*).

The chemistry of platinum and palladium is largely limited to the coordination compounds of these elements. The oxidation state of two is common for palladium, but the oxidation state of four occurs only in a few compounds, such as $K_2[PdCl_6]$. Palladium has an oxidation state of zero in the compound $K_4[Pd(CN)_4]$ (*10*). The oxidation states of two and four are commonly encountered in coordination compounds of platinum.

A few unstable compounds of platinum (III) have been reported, such as $M[Pt(CN)_4]$, which results from the oxidation of the platinum (II) compound with hydrogen peroxide (*38*). The compound $Cs_2[PtCl_5]$ was obtained by adding ice-cold chlorine water in small portions to a saturated solution of $Cs_2[PtCl_4]$ (*69*). Platinum (VI) derivatives, such as

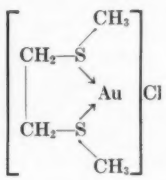
K_2PtO_4, have been obtained by electrolytic oxidation of hydrated platinum (VI) oxide dissolved in KOH solution (*68*).

Group IB

Soluble compounds of copper (I) such as $CuNO_3$ are apparently unstable. Sloan (*66*) prepared $CuNO_3 \cdot 2NH_3$ by reduction of $[Cu(NH_3)_4](NO_3)_2$ with copper foil in liquid ammonia. The colorless product was always contaminated with some copper and was oxidized very rapidly in air. Coordination with certain compounds stabilizes copper (I) to a marked extent, *e. g.*, $[Cu(NCCH_3)_4]NO_3$ (*52*), $Na_2[Cu(CN)_3]$ and $[Cu(SC(NH_2)_2)_2] \cdot NO_3 \cdot H_2O$ (*34*). Most other complexing agents stabilize copper (II) toward reduction, *e. g.* $[Cu(en)_2]I_2$. Copper (III) compounds of the type $M_5[CuTe_2O_{12}] \cdot nH_2O$ and $M_7[CuI_2O_{12}] \cdot nH_2O$ have been reported.

Silver (II) compounds are very strong oxidizing agents, but coordination compounds formed with α,α'-dipyridyl (*67*), *o*-phenanthroline (*27*), or picolinic acid (*3*, *14*) are remarkably stable. Silver (III) forms a compound with ethylenebiguanide (*60*) and another with periodate (*41*) similar to the copper (III) compound. Bailar (*1*) has reviewed the oxidation states of silver.

Although gold (I) compounds are fairly common, the only soluble compounds of gold (I) are coordination compounds, *e. g.*, $Au(CN)_2^-$ and (*34*):



The stabilization of gold (I) is particularly great with cyanide ion, since metallic gold can be dissolved in a solution of a cyanide salt in the presence of air. Most of the stable gold (III) compounds and all of the soluble compounds are coordination compounds, *e. g.*, $[Au(en)_2]Br_3$ and $[AuBr_2py_2]Br$.

CONCLUSIONS

It is unfortunate that one cannot list the factors contributing to stability of coordination compounds and the exact extent to which they affect the stability. However, there are many variable factors which are not thoroughly understood, and systematic and quantitative measurements of the stability of coordination compounds have only recently begun. The great activity in the field at the present time should yield much significant information within the next few years to help clarify the picture.

It is hoped that such studies will make possible some predictions concerning the stability and properties of coordination compounds before they are prepared. At the present time that is not possible except in very limited situations.

The quantitative data which have been obtained indicate that there is more regularity in the order of stability of metal complexes than has been supposed previously. Irving and Williams (*28*) pointed out that in the first long period (Mn, Fe, Co, Ni, Cu, Zn) the stability of complexes increases steadily to a maximum at copper; ligands may be ammonia, ethylenediamine, 1,3-propylenediamine, or salicylaldehyde. Zinc complexes are less stable than those of copper. The same general order is found for the complexes of dithizone and 8-hydroxyquinoline. More data are needed for the metals such as chromium (II) and for metals in other series. Many types of ligands should be investigated with particular emphasis on the cases of apparent specificity.

Studies concerning bond type and the electronic configuration of the central ion of coordination compounds should prove particularly fruitful. These factors seem to be particularly important in determining the stability of coordination compounds. Investigations concerning the stabilization of oxidation states, and particularly unusual oxidation states, should be of great value in extending and strengthening the systematics of inorganic chemistry.

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AN APPARATUS FOR STORING AND MEASURING SOLUTIONS OF LITHIUM ALUMINUM HYDRIDE[1]

CLYDE R. DILLARD
Tennessee A & I State College, Nashville, Tennessee

LITHIUM aluminum hydride has assumed an increasingly important role as a chemical reducing agent. More than 200 papers[2] concerning its reactions and those of related alkali-metal borohydrides have been published since Schlesinger and his co-workers announced their first results less than five years ago.[3]

Lithium aluminum hydride is prepared by the reaction of lithium hydride with aluminum chloride according to the equation:

$$4LiH + AlCl_3 \rightarrow 3LiCl + LiAlH_4$$

It is available commercially as an ether-soluble gray-white powder. The solubility of this compound in ethyl ether is 25 to 30 g. per 100 g. of pure solvent; however, it is usually desirable to use more dilute solutions.

The chief drawback to the use of lithium aluminum hydride is its extreme reactivity. Operations such as weighing, filtering,[4] or transfer of solutions must be done in an inert atmosphere or under vacuum. Moreover it is inconvenient to store large amounts of solution or to measure out exact aliquots from a stock solution. In several instances violent explosions have occurred when reactions involving lithium aluminum hydride were carried out in ether solution.[4,5]

It is the purpose of this paper to describe an appara-

[1] Presented before the Division of Organic Chemistry at the Southwide Chemical Conference at Wilson Dam, Alabama, October 19, 1951.

[2] *Chem. Eng. News*, **29**, 1203 (1951).

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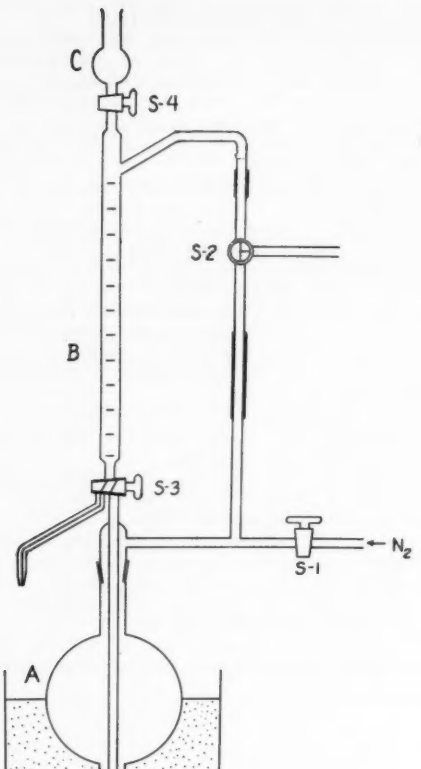


tus in which lithium aluminum hydride solutions may be stored for long periods of time without decomposition, and from which exact aliquots may be conveniently withdrawn.

The apparatus is shown in the figure. Its chief parts are a liter storage flask, *A;* a 25-ml. buret, *B;* an ether reservoir, *C;* two 1-mm. straight-bore stopcocks, *S-1* and *S-4;* one T-bored three-way stop-cock, *S-2;* and one oblique-bore three-way stopcock, *S-3*. The buret is made from a 25-ml. pyrex graduated pipet and is attached to the storage flask by means of a 24/40 ⅌ joint. The joint and stopcocks are lubricated with ether-resistant silicone grease. (Stopcock *S-3* should be lubricated regularly once a week.) For purposes of safety, the apparatus is set in a container of dry sand and the nitrogen inlet is connected by means of a T-tube to a mercury-filled pressure regulator (not shown in the figure).

The ether solution of lithium aluminum hydride is filtered directly into flask *A*, under a stream of nitrogen. Then the buret, *B*, is put in place with stopcock *S-1* open and stopcocks *S-2* and *S-3* turned so that nitrogen flushes out the buret. *S-1* is then closed and *S-3* is turned to communicate with the storage flask, *A*. A solution thus stored at room temperature under an atmosphere of nitrogen will remain clear and practically unchanged in composition for several months.

The procedure for withdrawing aliquots is as follows: The ether reservoir, *C*, is filled with dry ether and closed with a $CaCl_2$ tube. Stopcock *S-2* is turned to connect the buret with the atmosphere and nitrogen is admitted through *S-1* forcing solution up through *S-3* into the buret. *S-1* and *S-3* are closed in that order. A sufficient quantity of solution is withdrawn through *S-3* to fill the capillary delivery tube and the initial buret reading is taken. The desired aliquot is withdrawn into a receiver which may be simultaneously flushed with a slow stream of nitrogen. Then the final buret reading is taken and the excess solution is drained back into flask *A* through *S-3*. *S-3* is closed and a small amount of flush ether is admitted through *S-4* and withdrawn through the capillary delivery tube. This procedure is repeated several times to wash out the buret completely. Then, with *S-4* closed, dry nitrogen is blown through *S-1*, *S-2*, and out of *S-3*. Finally, *S-3* is turned to communicate with the storage flask, *A*, *S-1* is closed, and the solution is again left under an inert atmosphere.

SCIENCE TEACHERS' LABORATORY CONFERENCE

During the Summer Session of 1952 Duke University, with the cooperation of the North Carolina Department of Public Instruction, will conduct the second in a series of Science Teachers' Laboratory Conferences, July 21–25, inclusive. This Laboratory Conference is designed to give science teachers an opportunity to perform interesting and useful experiments which may be used by their own students.

There will be daily sessions conducted in the laboratories in the departments of biology, zoology, chemistry, and physics. Each session will be supervised by members of the science departments of the University and emphasis will be placed on individual participation. Ample opportunity will be provided for the exchange of ideas and the discussion of mutual problems that arise in the classroom.

For further details write to Dr. J. H. Saylor, Department of Chemistry, Duke University, Durham, North Carolina.

THE CONTRIBUTION OF STUDENT AFFILIATES TO CHEMICAL EDUCATION[1]

SISTER M. LAWRENCE O'DONNELL
Mount Mercy College, Pittsburgh, Pennsylvania

In order to write with any degree of conviction it is necessary that I write from personal experience. Hence most of this material will automatically center around Mount Mercy College Student Affiliates. Our chemistry department is very small, consisting of only three faculty members and an average of 12 chemistry majors. Yet we hope to equip our students in such a manner that they can rank equally with chemistry graduates from any and every undergraduate college. In order to accomplish our purpose, it is necessary that we broaden our scope much beyond the course content.

Prior to our A. C. S. affiliation, ever since 1936, a chemistry club existed on campus which met monthly to hear lectures from noted Pittsburgh chemists. Occasionally we substituted student papers for those of guest speakers. I select one aspect of the student activity during this time because it is one for which we were unable to obtain guest speakers. This aspect consisted of various phases of the relation of the Church to science. Students prepared papers on such subjects as "The attitude of the Church towards medicine in the Middle Ages," "The Galileo question," "Saint Augustine and evolution," "The Church and evolution in the twentieth century," and delivered them before the group.

In 1944 we organized our chapter of Student Affiliates. We hoped that this affiliation would complement our teaching program and increase the efficiency of our department, thereby supplying for the deficiencies inherent in a program with a limited staff with limited time. Each year with increasing developments we are more convinced of the soundness of our program. The Chapter meets twice a month on the first and third Tuesday at 8:00 P.M., having the advantage of the total faculty of chemistry teachers of the Pittsburgh area as guest speakers. Those speakers are relatively easy to obtain. Chemistry professors from the four universities or colleges, fellows from Mellon Institute, and leaders in industrial chemistry are glad to air their pet hobbies or special research interests to a college audience. Aside from their actual enjoyment in talking about their own subjects, they do not scorn the publicity attached. They are always given adequate recognition in the *McAuleyan* (the college publication), the *Crucible* (the publication of the Pittsburgh section of the American Chemical Society), and the final report of the student affiliates to Washington. Their lectures bring their organizations before the chemical public, secure for them the approbation of their superiors and a feeling of satisfaction in their own personal contribution to chemical education. We have had such men for speakers as the chairman of the Pittsburgh section of the A. C. S., the chairman of the Employment Bureau of the A. C. S., and heads of departments of chemistry from the other Pittsburgh universities and colleges. Our alumnae, too, have returned to tell us of their experiences in different divisions of the chemical profession. The alumnae never fail to make a favorable impression upon the undergraduates. They add a sense of reality and lend a personal touch. They convey the attitude not only that it can be done, but that *you* can do it. "We are a proof of it. We are doing it; so can you." The alumnae "belong," and hence the personal or familiar touch.

Incidentally, we are rather particular in our choice of speakers at Mount Mercy. We aim for two requisites: a good speaker and a chemical subject. By that I mean that we don't ask just any chemist to talk about just anything for the sake of having a speaker. We want to be fairly certain that our students will be interested and inspired. When students remain at school until evening or return from home at 8:00 P.M. after a day's work, we want something more than activity for activity's sake. Nothing will discourage a student more quickly than boredom. The type of speaker that the students most enjoy is one who has all the factual material they are expecting, who presents it with demonstrations, whose illustrations are amusing, who does not "talk down" to them, and who is not "stuffy." This man will be remembered and quoted and discussed in and out of season. He represents their idea of "a chemist." He is their future colleague. Professional association with such as he is their idea of the chemical profession. We have had other speakers, not too frequently, thank goodness, who have had the opposite effect on the students. These were men who knew their material, knew what they wanted to say, and said it fluently, but who depressed the students. They "talked down" to them, not only from the height of their immense intelligence and immense knowledge but even from the height of their superiority as a man. Fortunately this type is more easily forgotten because he is seldom discussed or quoted.

All the activities of the student affiliates help to develop professional consciousness in the students, but the meetings with the preparations involved furnish a special opportunity for this development. The president is responsible for making all arrangements with the

[1] Paper read before the Chemistry Section of the Pennsylvania Chapter of the Catholic Round Table of Science at the meeting at Scranton University, April 28, 1951.

speakers. She invites them, decides the date and place of the lecture with them, interviews them, and introduces them at the meeting. The secretary, or a member with journalistic tendencies, makes a report of the talk and of the meeting to the *McAuleyan*. Very often a social hour follows the meeting and at that time the students talk informally with the speaker. While entertaining informally, they are at the same time being trained in the responsibilities of hostesses and learning to "talk up" to the professional level demanded by the occasion.

Once or twice a year a chapter in the Pittsburgh area plays host to the other chapters for an evening lecture and a social hour. In this way the students meet their future professional associates, talk shop and non-shop over cookies and punch or while dancing to the tune of the juke box. These joint meetings are very profitable. They accomplish easily and gracefully what the teacher has a difficult time doing without conveying the idea of pride or prejudice. They stimulate the students either to reach the standards of those better qualified than themselves or to exhibit with pride their own accomplishments. The students are quick to recognize deficiencies by comparison, and eager to rank with the best.

The curriculum, as is to be expected, provides no time for tours of chemical plants or industrial laboratories. And yet the first-hand information and practical knowledge attained in such tours are an invaluable complement to the course material and cannot be attained elsewhere. These tours can be made under the sponsorship of a professional organization. In our case, not only the student affiliates attend, but a general invitation is issued by bulletin board announcements, and interested students from related departments are welcome. The Chapter makes at least one such trip during the year. To mention a few among many, we have visited the Westinghouse Van der Graaff generator and laboratories, the University of Pittsburgh cyclotron, Mellon Institute, Gulf Research Laboratories, and Heinz Plant.

To send out a chemistry major into industry or other chemical fields with no idea of research is to send out one with mediocre preparation. Chemical competition among men is great. A woman must be equipped with the best that an undergraduate course can give if she is even to enter such competition. Therefore, we attempt to form a research attitude through the student affiliate seminar program. The students are encouraged not only to engage in research, but to present their results of that research before the affiliates of the area and to compete for awards.

One of the most important functions of our student affiliates is the seminar course. All sophomore, junior, and senior chemistry majors are required to take seminar for three years. This course consists of a double program: one hour class per week and one afternoon of research. At the class hour the students present papers, each student contributing at least one hour each semester. This year the course is devoted to Pittsburgh industries; last year we studied advanced inorganic chemistry. We plan courses in instrumentation and in history of chemistry in some future years. In our research program, the sophomores usually begin with a literature search to acquaint them with the chemical literature and to teach them how to use it, particularly *Chemical Abstracts* and Beilstein. Incidentally, this literature search serves not only to teach the student but to collect data for the research program of the department. The upper-classmen do laboratory research. One group at present is trying to prepare esters by a method not yet in the literature. One senior is making some studies on pyrrole by means of the new Beckman spectrophotometer. Another senior has spent an average of 12 hours a week investigating the toxicity of certain chemical groups on duckweed. This girl presented a paper at the student convention this year. Results of earlier cooperative work on herbicides were presented by Dr. Fromm before the Division of Agricultural and Food Chemistry of the A. C. S. at the Atlantic City meeting and will be published presently in *Botanical Gazette*. Our major difficulty at Mount Mercy is lack of cooperators—not that our students fail to cooperate, but that there are not enough of them nor is there enough time to tackle the many problems that arise.

The highlight of our chemical year is the student convention. This is modeled on the A. C. S. convention but lasts only one day. It, too, has a short but interesting history. Three years ago Sister Mary Dorcas of Seton Hill College sent letters to St. Vincent, Duquesne, and Mount Mercy asking our opinion of the feasibility of a student meeting and inviting us to Seton Hill College to present our papers. The colleges approved of the idea and we had our first student convention. All so heartily agreed on the success of the venture that the next year Duquesne University invited all in the Pittsburgh area to participate. It, too, was a real success. This year Mount Mercy held the meeting. We asked for sponsorship by the Pittsburgh Section of the A. C. S., which not only agreed to sponsor, but offered 50 dollars for prizes. At this meeting, Dr. R. R. McGregor, the past chairman of the section, was the guest speaker, and the chairman-elect, Dr. E. O. Rhodes, an industrial chemist, was chairman of the award committee. The papers, nine in number, were limited to undergraduates and presented by students from Bethany College, Carnegie Institute of Technology, Duquesne University, St. Vincent College, Seton Hill College, and Mount Mercy. About one hundred students attended from the five Pittsburgh colleges, Bethany, College of Steubenville, Geneva, St. Francis, St. Vincent, Seton Hill, and Youngstown Colleges. The meeting was followed by a social hour for the students, a faculty meeting for sponsors, and a meeting of the award committee. It ended with the announcement by the chairman of the award committee of the winners of the awards.

We feel that the activities resulting from student affiliation with the A. C. S. contribute a large share to the efficiency of our department and enable us to give our students a chemical education second to none on the undergraduate level.

PHYSICS AND CHEMISTRY FOR GENERAL EDUCATION

KENNETH M. SIMPSON and JOHN W. SUTTON
Santa Barbara College, University of California, Santa Barbara, California

So many descriptions of courses designed for the general education of the nonspecialist have appeared since the last war that one must surely offer an apology for presenting another. The apology in this case takes the following form. The problem is an important one, so important that it is unreasonable to overlook any attempted solutions which have elements of novelty or practicality in them. It is, indeed, the practical advantages of our course which we wish to stress. For many a noble and needed experiment has failed by demanding too much of the experimenter, by demanding an experimenter of such rare gifts that everything hangs on one man, who turns out, quite frequently, to be made of flesh and blood. We have made, we think, a fair start on a course for the general student which is workable with staffs as they are in small or moderate-sized colleges and universities and which yet provides a framework, a favorable atmosphere, in which to achieve many of the objectives formulated by those impressed by the need for nonprofessional education in the sciences.

This course has been, from the beginning, something of a cooperative effort. It came into being as a result of a reorganization of the entire college curriculum so as to implement the University of California's policy of specializing in undergraduate education at its Santa Barbara campus. As part of the program, the Physical Sciences Department was asked to offer a year course, Physical Science 1A-1B, especially designed for the general education of the nonscience majors.

Our whole staff, more than a dozen chemists, physicists, and geologists, found they could agree on three principles from the start. The first, a negative principle, was that we should not offer just another survey course. The interesting fact is that, although many a telling argument can be made against the survey type of course, none seemed demanded. This distaste for the survey appears to be nearly universal among scientists and constitutes almost sufficient ground for not attempting such courses, whether the distaste be objectively justified or not. A positive principle was that the course should contain solid science. Another was that there should be laboratory work. Again, in its insistence on these two points, this department does not strike us as atypical.

Starting from these conditions the decision was made to limit the course, so far as fields of science were concerned, to physics and chemistry. This decision was based on a number of arguments, not each of which, to be sure, was equally persuasive to all staff members. We felt that it was very desirable to have one man give all, or nearly all, the lectures in any one section. Hardly anyone was available who could pretend to any sort of competence in physics, chemistry, and geology. There were, on the other hand, three physical chemists with good training in physics and five physicists who knew a fair amount of elementary theoretical chemistry. This situation again appears not unusual. It is important that a number of men be available to give the lectures in the course, not only because more than one section may need to be given but also because any man teaching a required or semi-required course year after year is liable to go stale on the job. Moreover, as this is a laboratory course, several staff members were needed to teach the laboratory sections.

There are theoretical advantages to emphasizing the fields of physics and chemistry. In the course of our discussions the view developed, and was accepted by many of us, that the division of the sciences into those concerned with things that are alive and those concerned with things that are not alive is more neat than illuminating. A more fundamental division should be one based not on the objects investigated but on the kind of investigation. Physics and chemistry embody the exact mathematical analytic aspect of science more perfectly than any other discipline. Here the descriptive aspects are at a minimum. Here the laboratory, the art of measurement, the use of experiment, have been carried further than anywhere else. Nowhere else does one find the organic unity achieved by great conceptual schemes so well displayed. If contrast with the biological sciences is desirable in a course in the physical sciences, here is where it is sharpest.

One may urge, further, that physics in particular was the first natural science to reach maturity, that it has been historically the paradigm for all others, even, whether foolishly or not, for the social sciences. There appears to be a kind of hierarchy of sciences (not, God forbid, of scientists): chemical properties and behavior are explained by physical concepts and theories, geology and astronomy are explained in terms of physics and chemistry, and, as a working hypothesis at least, biological behavior is explained in terms of chemical ideas. From the point of view of what is fundamental, a good case can be made for concentrating on physics and chemistry.

It is obvious that many topics are difficult to allocate

between physics and chemistry. Indeed, such a division is not too useful for understanding these disciplines, however important it may be to workers in some speciality. The problem still remained as to how topics should be arranged and transitions managed. (Query: How is the transition made from, say, Heat to Optics in a standard physics text. Answer: By starting a new page. But there no one worries about what makes these subjects both physics.) Profit of a not clearly anticipated sort has accrued from the decision to do both physics and chemistry. First, it has made it more difficult for us to fall into the familiar ruts. Second, it has turned out that the physicists like doing some chemistry and the chemists like the physics. We have all had to learn something.

It is a truism that one should have the ends in mind before designing the means. But he should also know what means are available before choosing ends. We have tried to do both in a back-and-forth, give-and-take sort of way. As a self-disciplinary measure and *aide-mémoire* in giving lectures, we have tried to formulate both what we are aiming at and what we are not aiming at. These aims are here listed and classified, but it is important to be aware in reading them that instructors in the course are not bound to emphasize each to a predetermined degree, for, we believe, the peculiar genius of one instructor will be such that he can achieve this, of another that he can achieve that, and each should do what he can do well.

Our objectives may be put under three headings: (1) To teach some solid subject matter. (2) To give some understanding of science both as a body of knowledge and as an activity. (3) To inculcate certain attitudes. Concerning the first of these there is remarkable agreement among the members of our staff, although the reasons different men have for supporting the aim are diverse. "Solid subject matter" is, of course, a comparative term. The typical college student simply cannot survive in a modern preprofessional course in these fields. What is meant here is that we do not want to give a course in which too many topics are treated too superficially. That is, not only should the course not be a survey of all the physical sciences but within the chosen fields of physics and chemistry the so-called block and gap system should be used.

To know what one does not want to do is almost as important as knowing what one wants to do. We do not aim to make a man feel at home among modern gadgets. (What scientist understands them all? We all work with mysterious black boxes.) Nor is the aim to enable the future housewife to cook scientifically or fix door-bells or television sets. Nor is our aim to entertain for an hour with a rapid succession of undigested demonstrations.

What then are the excuses for teaching solid science? Briefly, they are: That the stuff is intrinsically interesting; that these achievements are some of the brightest jewels in Western Civilization's battered crown; that one area of physical knowledge, atomic and nuclear physics and chemistry, has such great contemporary significance that the educated man should have some comprehension of it; and, finally, that any attempt to show the methods of these sciences, science as an activity or process, is hopeless without real illustrative material. Probably those who object to courses "about science" are making this last point.

Under the second class of aims we include study of the methods and the logic of these sciences—for example, the manner in which a crude notion such as hot or cold becomes by a process of successive approximation a precise physical concept—or the use of models, such as the particle or wave model in constructing a theory. Here, too, we include the sort of thing Conant calls the "tactics and strategy of science." We have used his case-history method only sparingly; it is not essential to his main points.

We have also included, as contributions to the understanding of science as a human activity, discussion of the interrelation of science and technology, the question of freedom of inquiry, and the changing methods of supporting the scientific enterprise. The discussion of the steam engine, for example, has been included not only because it is germane to the work on heat but also because it permits the point to be made that in the eighteenth century it was possible for a fairly elaborate technology to develop almost completely without the aid of science, and the further point that this is, in general, no longer true.

As for the third of the major classes of aims, there are two sorts of attitudes we believe worth encouraging. The first could be called the scientific attitude—a state of mind conditioned by the knowledge that the world is as it is rather than as one wants it to be, and by the faith that any genuine question about the world will ultimately yield to patient and inspired inquiry in accordance with the methods of science. The second we would describe as a friendly attitude toward science. There are so many signs of the growth of an unfriendly attitude toward science—perhaps as an irrational reaction to rapid change or atom bombs—that we think it not illegitimate to attempt, in a small way, to counteract this development.

In the working out of a syllabus of topics each lecturer has had a rather free hand. The reasons for freedom are the obvious ones that instructors do better work when allowed to exercise choice and initiative and that experimentation is desirable. One such outline of topics (it happens to have been used, with minor modifications, more than any other) is briefly sketched below. Again, almost more important than noticing what is included is noticing what is left out. Examples of standard topics omitted are: vectors, statics, geometrical optics, rotary motion, alternating currents, most of sound, elasticity, and the systematic study of the chemical properties of the elements. The study of vectors, to single out one case, is no doubt interesting and valuable, but as with the charity boy in "Pickwick Papers" when he came to the end of the alphabet, why should one go through so much to learn so little? Of the topics our list includes, many are normally given

Lecture Topics

First Semester:

(8[a]) Galileo and the beginnings of modern science. The Newtonian synthesis.
(2) The development of the gas laws.
(8) Fundamental chemical concepts and laws and chemical atomic theory.
(4) Heat and energy—the steam engine, heat, potential and kinetic energy, mechanical equivalent of heat, conservation of energy.
(2) Kinetic theory.
(3) Development of the periodic law. Structure of atoms.

Second Semester:

(10) Chemical bonds, ionization, chemical energy, kinetics and equilibrium, organic chemistry.
(7) Static electricity, electrolysis, Ohm's law and the heating effect, electromagnetism.
(2) Wave motion.
(8) Atomic physics—X-rays, radioactivity, the Bohr-Rutherford atom, photo-electricity, isotopes, artificial transmutation, atomic energy.

Laboratory Experiments

First Semester:

1. Review of algebra.
2. Hooke's law.
3. Simple pendulum.
4. Freely falling bodies.
5. Gay-Lussac's law.
6. Chemical elements and compounds.
7. Atomic weight of copper.
8. Molar volume of oxygen.
9. Molecular weight by Dumas' method.
10. Preparation and properties of hydrogen and oxygen.
11. Specific heat.
12. Mechanical equivalent of heat.
13. Halogens and alkali metals.

Second Semester:

1. Acids, bases, and salts.
2. Reactions of ionic materials.
3. Elementary qualitative analysis.
4. Factors influencing rates.
5. Synthesis of ethyl acetate.
6. Electric cells and electrochemistry.
7. Electrolysis.
8. Volts, ohms, and amperes.
9. Heating effect of an electric current.
10. Motor effect of the electric current in magnetic field.
11. Wave motion and resonance.
12. Microscope and telescope.
13. Wavelength of light by a grating.
14. Microwave demonstration.
15. Radioactivity demonstration.

[a] Approximate number of lectures given.

in both first-year physics and first-year chemistry courses; for example, the gas laws, kinetic theory, the concepts of heat and energy, all of the elementary atomic theory, and, of course, the relearning of elementary algebra, a subject which seems to be dying of neglect in our high schools. The majority of our students have had neither physics nor chemistry in high school. Those who have had one of these find, in general, that the approach is sufficiently different to take the curse off the repetition.

This is a year course, with two lectures, one two-hour laboratory period, and one hour for discussion sections. We have found that the sternest self-discipline is required to keep ourselves from trying to include too much. Indeed, we have not been stern enough and hope in the future to use the knife more ruthlessly.

Most physical-science courses for the general student have omitted the laboratory, primarily because of expense. So long as there is no really persuasive way of estimating the value of the laboratory as compared to the lecture, science staffs can hardly do more than record their expert opinion that laboratories are worth what they cost. We have, however, done much to make them cheaper. The periods are limited to two hours, thus permitting a room to be used four times a day. Although all of the experiments are run on the even-front principle, the apparatus has been kept cheap except in those cases where the experiment is of the demonstration type. Cheapness and simplicity of apparatus has the inestimable value of permitting changes from year to year. Although elementary chemistry has never suffered seriously from the need of expensive equipment, elementary physics laboratories are too often confined by their own heavy investments. We have deliberately designed our electricity experiments, for example, so that the same rheostats and meters can be used throughout. In chemistry, experiments have been adapted for the use of semimicro technique. We find that this helps make it quite feasible to use a physics laboratory for the chemistry or vice versa, thus making it economically possible to mix physics and chemistry experiments in the same semester. For the physics experiments the apparatus (either 8 or 16 sets) is set in the laboratory for the entire series of sections. We have also found it a simple and successful practice for most of the chemistry experiments to have equipment for the whole week set out in 16 to 18 trays. Each student uses the equipment in a tray and then replaces it for the one who will succeed him in the next section. By this means we avoid the necessity of expensive tables or cabinets containing a drawer for each student enrolled in the course.

Mimeographed directions are furnished the students. Data are taken, curves plotted, observations recorded and queries answered in a bound cross-section paper notebook. We try to have the students complete their write-up in the laboratory and most of the checking of the written work and computations is done there. The custom of writing long elaborate reports a week after the work is done is the most unprepossessing and least rewarding feature of the traditional laboratory course so far as nonprofessionals are concerned. Reading such reports is making poor use of faculty time and, in colleges at least, good student readers are hard to find.

It is the policy here to have faculty members conduct the laboratory periods. Not only are competent student teaching assistants hard to come by in the absence of a large graduate school, but also we find that nonprofessional students require more skillful aid and more of it than do students in the major courses. Moreover

it is probably uneconomical to invest in laboratories at forty dollars per square foot, the going price in California, and then try to save a few dollars on teaching staff.

The laboratory work has a variety of functions. One of the most important of these is to show the student that material things have a diabolical cussedness which the scientists must somehow subdue. One is to drive home the point that a large part of scientific method consists in the use of innumerable and almost unclassifiable techniques. (This is one of the chief reasons why a skilled physicist is not automatically a skilled biologist or economist, as he would be if "the" scientific method were one single thing.) Another function is to impart some degree of sophistication in regard to accuracy of measurement. Still another is to make vivid the tremendous gap between the concrete experimental facts, say, of the gas laws and the explanatory kinetic theory. Some of the experiments have as a primary purpose the clarification of difficult ideas. Some demonstration experiments have been chosen for their elaborateness and timeliness—for example, the one on radioactivity using a scaler. Two or three, alas, are there for want of something better, but, we expect, not for long.

Some major difficulties remain unsolved. One is the problem of textbooks. We tried one survey text when the course was being given on a pilot scale and found that when we had omitted geology, meteorology, and most of the astronomy the rest was too far condensed and inadequate. We have since then used a physics text plus a chemistry text, but do not find this a very satisfactory answer even when they are treated as references. We find, for example, that no texts treat the subject of reaction rates as completely as we want to except those which, like Pauling's, are too formidable to put in the hands of these students. (Suggestion: It would be of great service to all instructors employing the "block and gap" system—a system which seems likely to meet with increasing favor for general education courses—were there to be published a set of "blocks," skillfully edited and coordinated. These might well be in pamphlet form, like the case histories now coming from Harvard.)

No books we have examined, except Taylor's "Physics, the Pioneer Science," and the Harvard Case Studies, pay more than lip service to scientific method, the logic of the subject, its "tactics and strategy" or social relations. Textbooks still carry weight with young students, and orthodox texts have proved for us almost worse than no texts at all.

Although suitable texts are likely to be forthcoming in the near future, the mental set brought by students to a physical-science course is a difficulty that will be long with us.

We have in this course students from all four college years. We are inclined now to think that the more general aims of the course can best be achieved with more mature students. It is a mistake to hold that general education should precede special or technical education. A boy of 16 can learn calculus or chemistry perfectly well and is often eager to, but he is seldom willing to reflect on the nature of mathematics and chemistry or what makes them grow. We believe it likely that with the exodus of the survey there will gradually come a shift of these general-education courses to the last two years of college.

Another unsolved difficulty is that of determining whether we are achieving our aims. In so far as these are traditional, traditional testing does it, but to measure the success of our other aims requires new techniques which we do not yet have. The size of the classes almost precludes the use of essay-type examinations, which might, if skillfully put and read, tell a good deal. Objective tests must be the answer, but so far we cannot claim to have made much progress in this direction. In the meantime we have our personal impressions, gained from contact with students, to encourage us. And should we fall even a good way short of our goal, we can remember that the dictum that anything worth doing is worth doing well should have as its running mate the wise saying of Samuel Butler, that anything worth doing is worth doing badly.

EASTERN COLLEGES SCIENCE CONFERENCE

The Science Departments of Pennsylvania College for Women announce the Sixth Annual Eastern Colleges Science Conference to be held on their campus April 3, 4, and 5, 1952. This conference will include over 300 eastern colleges offering the various branches of science. More than 500 undergraduates will represent their colleges.

The theme for the conference will be "Science in an Industrial Era" and will center around the industries of Pittsburgh. Tours will be conducted through Westinghouse Electric, a steel mill, H. J. Heinz Company, Buhl Planetarium, Western Psychiatric Hospital, Carnegie Museum, and Mellon Institute of Industrial Research. The program will also include exhibits, social functions, speeches by nationally known scientists, lectures by specialists in various fields of science, and most important of all, the presentation of undergraduate student papers and the exchange of student ideas.

A CONSTANT-TEMPERATURE BATH FOR WARM CLIMATES

W. M. LOVELESS and MADISON L. MARSHALL
Texas State College for Women, Denton, Texas

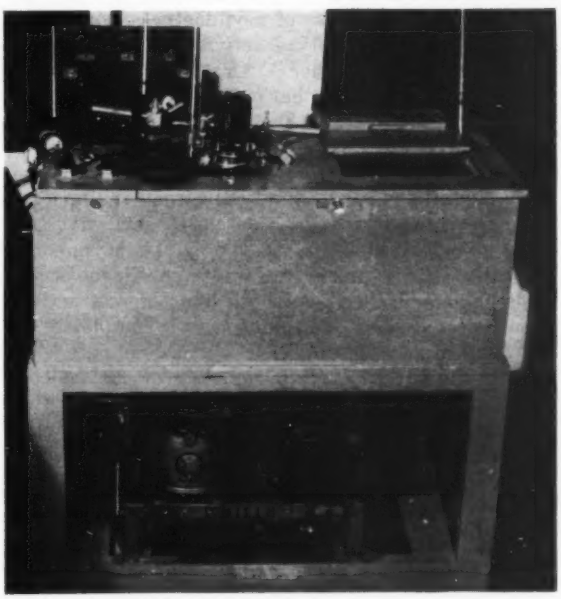
CHEMICAL development in the southern states poses a new problem in temperature control. In some areas the maximum daily temperature exceeds 35°C. for days at a time. For weeks, and sometimes for months, hydrant water may remain above 25°C. The experimenter is forced to make his studies at temperatures considerably above this or he must resort to some type of refrigeration.

Although some buildings are air-conditioned there is a definite need for a simple water bath with a cooling arrangement that has sufficient maneuverability to be easily moved from room to room or from building to building. The conventional water bath with cooling coils leading through an ice box or modified refrigerator is sometimes used. Such an arrangement is capable of giving good temperature control but is unduly space-consuming and generally difficult to move once it is in operation. The authors designed and built a compact constant-temperature water bath for instructional use in physical chemistry but which has a wider range of application.

The bath consists of two tanks, one within the other. The outer tank is 40 in. long., 18 in. wide, and 16 in. deep. It is filled with water which serves as a refrigerant. It is made of wood mounted on a wooden frame, supported on castors and lined with stainless steel, and is light enough so that even when filled with water the entire assembly can be moved from place to place in the laboratory.

The bath assembly is designed in such a manner that one end of the larger tank is used for precision temperature control while the other end is used for the storage of solutions or for temperature regulation of standard solutions during their preparation. For precision temperature control a Sargent constant-temperature unit is placed in the inner tank (a 20-liter jar) surrounded by the refrigerant. Temperature in this tank is maintained over long periods of time with little attention to within ±0.01°C. For a period of a few hours the variation is considerably less, varying no more than a few thousandths of a degree. The temperature is determined by a mercurial thermometer calibrated by the U. S. Bureau of Standards.

The coolerant in the larger tank is warmed by a simple type of immersion heater. The coolerant flows through copper tubes which are attached to the inside walls of the tank, traversing the length of the tank ten times before returning to the compressor. The refrigeration unit is a $^1/_4$-horsepower size of the type supplied by a local refrigerator concern and requires 460 watts for operation. Water is circulated by means of a water pump discharging through a copper tube running the length of the tank, closed at its extremity but perforated with holes along its entire length. Both the heater and refrigeration units are controlled by Fenwall expanding type electric thermoregulators. Maximum sensitivity for this type of regulator is listed as ± 0.1°C. Some difficulty was encountered with the above arrangement until relays were inserted between the thermoregulators and the heater and refrigeration units. A terminal box which eliminates bare wires and provides convenient grouping of switches completes the assembly.



The Constant Temperature Bath

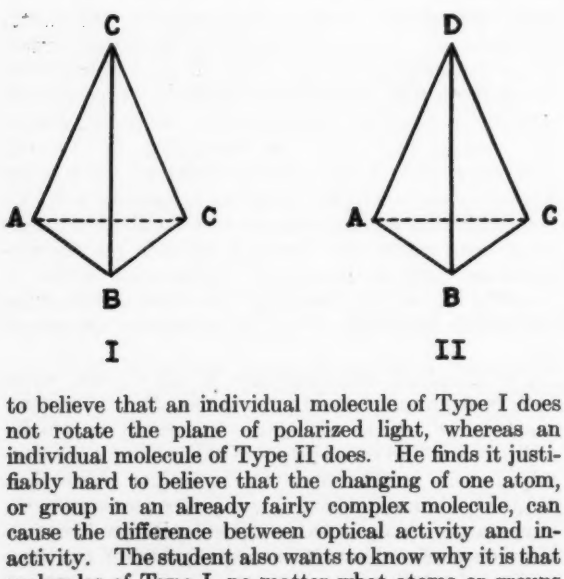
For precision measurements at 25°C. the outer bath is maintained at approximately 20°C., the temperature at which volumetric apparatus is usually calibrated. The temperature of the coolerant is maintained easily at this value with variations of less than ±1.0°C. Volume changes in water accompanying temperature changes within these limits are less than those encountered in volumetric procedures in the preparation of solutions.

THE CAUSE OF OPTICAL INACTIVITY

DWIGHT FAY MOWERY, JR.
Trinity College, Hartford, Connecticut

It is possible to pick up many introductory, or advanced, organic chemistry textbooks and find one of the following statements given as a reason for optical activity or inactivity: "Loss of asymmetry automatically involves the loss of optical activity. The two properties disappear together." Or, "The crux of the theory presented here is that optical activity rests upon molecular asymmetry. The theory has been completely successful in the face of thousands of tests." Or, "The fundamental postulate of the theory is that this isomerism is to be found only when the molecule can exist in two forms which are mirror images of each other. Such a molecule is said to be asymmetric. Optical activity is a property of an asymmetric molecule." And, finally, "Experience shows that optical activity is only produced when the molecule is completely unsymmetrical in structure. All chemical changes which remove the cause of the asymmetry of the molecule result in a disappearance of the optical activity. One is therefore led to the conclusion that the capacity possessed by many substances of rotating the plane of polarization of light has its origin in unsymmetrical molecular structure."

It will be noted that in all cases the connection of optical activity and molecular asymmetry is purely empirical. This would not be so bad in itself if it did not lead to an incorrect picture of optical activity which defies logical explanation. The student is led



to believe that an individual molecule of Type I does not rotate the plane of polarized light, whereas an individual molecule of Type II does. He finds it justifiably hard to believe that the changing of one atom, or group in an already fairly complex molecule, can cause the difference between optical activity and inactivity. The student also wants to know why it is that molecules of Type I, no matter what atoms or groups are represented by *A*, *B*, and *C*, are always optically inactive, whereas all molecules of Type II are always optically active. The answer is very simple, and is found in almost any paper on the theory of optical rotation. In all these theories (*1*) a summation of the rotations of all the individual molecules, oriented in all possible random positions, is carried out, and in the case of molecules with a plane or center of symmetry, this summation produces a net rotation of exactly zero. The physical picture of this summation is described in a paragraph written by Noller (*2*) in 1947 and, unfortunately, not incorporated, to the author's knowledge, in any of the newer textbooks. Referring to the compounds pictured, containing a single carbon atom, Noller says: "If two atoms or groups are alike, the random orientation of the molecules will provide each molecule with a mirror image so that any rotatory effects of the individual molecules would cancel. Therefore the only requirement for optical activity is that two like molecules cannot be orientable in such a way that they are mirror images of each other or in other words that the mirror image of a molecule cannot be superimposable." It is evident that if a molecule has a plane, or center, of symmetry, it is identical with its mirror image, and, in any solution of sufficient concentration for optical measurements, there will be, for every molecule oriented in a given way another oriented as a mirror image of the first so that the resultant rotation of the solution will be exactly zero. The rotations produced by the individual molecules will be "externally compensated" in the same manner as for a *d,l*-mixture.

The erroneous impression given by textbooks, of individual molecules which are optically inactive because of a plane or center of symmetry, is probably caused by attempting to explain the conditions for optical activity rather than those for optical inactivity. This is due to the fact that optically inactive compounds were known long before, and in much greater numbers, than optically active ones. If optically active compounds had been discovered first, and then occasionally an optically inactive one had come to light, it is very likely that the correct explanation of optical inactivity would have been arrived at earlier.

This picture of optical inactivity can also be applied to meso-tartaric acid, where it is seen that the inactivity is probably due principally to "external compensation." The "internal compensation" in this case would be limited to exactly "staggered" molecules or "eclipsed" molecules, which are oriented with their planes of symmetry perpendicular to the path of the light (*3*, *4*).

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A NEW EXPERIMENT ON REACTION RATES IN GENERAL CHEMISTRY

GORDON G. EVANS
Tufts College, Medford, Massachusetts

A SURVEY of several laboratory manuals in general chemistry shows that three experiments are in use to illustrate the effect of concentration on reaction rate. These are: (1) the rate of evolution of hydrogen by the reaction of a metal and an acid, (2) the rate of liberation of sulfur from an acidified thiosulfate solution, and (3) the "iodine clock." None of these can be considered ideal for the purpose. In the case of the first, only one of the reactant concentrations can be varied; in the case of the second, the kinetic dependence is not a simple bimolecular one,[1] and the opacity of the resulting sulfur suspension, by which the rate is usually measured, is dependent on certain factors in addition to the concentration of sulfur.

The "clock" reaction, though apparently the most popular, is subject to the most serious objections, some of which have been pointed out by Hered.[2] It should also be noted that the time elapsed is that required for exhaustion of the sulfite ion, and therefore it is not possible to use the reaction to show dependence of the rate on the sulfite concentration; in those instances where the reaction is complete sooner at a higher initial concentration of sulfite, another factor (acid catalysis) is operating which is concealed from the student.

The reaction between persulfate ion and iodide ion appears to be free from all these objections. The reaction is bimolecular,[3] the concentrations of both reactants can be varied, and the effect of depletion of the reactants can be minimized by observing only the first part of the reaction. The time required for the development of a predetermined depth of color serves to establish the reaction rate. Concentrations which have been found convenient are 0.1 *M* $(NH_4)_2S_2O_8$ and 0.2 *M* KI. If a solution containing iodine (0.001 *M*) and potassium iodide (0.03 *M*) is used as a color standard, about two minutes will elapse from the time when equal volumes of the reagents are mixed until the color of the reacting mixture matches that of the standard. The effect of concentration can of course be shown by diluting one or the other reagent before mixing. In the experience of the author and his colleagues, students' results in this experiment have been generally quite satisfactory.

Two drawbacks to the experiment have become evident. The first is the cost of the reagents; unfortunately, the solutions cannot be much more dilute than is stated above without prolonging the reaction time inordinately. However, the cost may be offset to some extent if the students' mixtures are collected in a large jar; the further progress of the reaction leads in a few days to the exhaustion of the potassium iodide and the deposition of crystals of iodine, which can be recovered. The other drawback is the difficulty of observing the point of exact matching of the colors. Students have a tendency to anticipate this point, and continuous close watch of the test tubes fatigues the eye; it is well to instruct that the tubes be compared every 15 seconds (not oftener) and that observations be continued until the reaction tube is definitely darker than the standard, whereupon the time when the colors matched may be determined by interpolation.

Finally, it must be noted that the ammonium persulfate must be pure and the solution freshly prepared; otherwise an *immediate* liberation of iodine may take place, rendering rate measurements useless.

ACKNOWLEDGMENT

The author wishes to express his gratitude to Professor Robert D. Eddy for his cooperation in testing the experiment.

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COMBINING HALF-REACTIONS AND THEIR STANDARD ELECTRODE POTENTIALS

SIDNEY I. MILLER,[1]
University of Michigan, Ann Arbor, Michigan

FROM his first year of chemistry through his analytical courses the student uses an activity series and standard electrode potentials before he understands the free-energy relationships involved. At all levels the usefulness of such a series in the teaching of chemistry has long been recognized. To increase the value of such a table of standard electrode potentials a new method of combination of half-cell reactions is proposed.

The proposed method of combining half-reactions and their corresponding electrode potentials is based on the method of volt equivalents (*1*). With the early introduction of this method as soon as half-reaction potentials are presented, the later transition to the free-energy concept should be natural and logical. No claim is made for the originality of this approach, but if the difficulties inherent in the alternative method are generally recognized, then an important aim of this paper will have been satisfied.

The data in the table on page 141 and the conventions followed are taken from W. M. Latimer's "Oxidation Potentials" (*1*). The relationship

$$-\Delta F = nE\mathfrak{F} \qquad (1)$$

in which ΔF is the free-energy change, E the oxidation potential, $\mathfrak{F}$ the faraday, and n the number of equivalents changed, will be referred to in the discussion. The number of volt equivalents, nE, is directly proportional to $-\Delta F$ and is commonly used instead of it for the calculation of E from the combination of any two half-reaction potentials (*1*). It is not suggested, however, that "free energy" need necessarily be brought into class discussion at the elementary level.

In the combination of half-reactions and half-reaction potentials two working "rules" or conventions are often followed (*1*). The first of these states that a *complete* chemical reaction is obtained by subtracting one half-reaction from the other; the standard e. m. f. for the complete reaction is obtained by subtracting the corresponding standard electrode potentials:

		E°	*Volt equivalents*
(I)	$3\,(Mg \rightleftharpoons Mg^{++} + 2e)$	2.34	14.04
	$2\,(Au \rightleftharpoons Au^{+++} + 3e)$	−1.42	−8.52
	$3Mg + 2Au^{+++} \rightleftharpoons 3Mg^{++} + 2Au$	3.76	22.56

This rule is found in a variety of textbooks (*2*, *3*, *4*, *5*). For the early courses the presentation is, of necessity, arbitrary. Thus, the student must be cautioned against multiplying the first e. m. f. by 3 and the second by 2 to get the e. m. f. of the combined cell of example I. In qualitative and quantitative analytical courses the more general Nernst relation (*5*),

$$E = E^\circ - \frac{RT}{n\mathfrak{F}} \Sigma\, \gamma_i \ln a_i \qquad (2)$$

which shows how the half-cell potential changes with concentration, may be given (*4*). The γ_i are coefficients and the a_i are activities of the species involved in the half-reaction. The rule of combination is then given as:

$$E_1 - E_2 = E_1^\circ - E_2^\circ - \left(\frac{RT}{n_1\mathfrak{F}} \Sigma\, \gamma_{i'} \ln a_{i'} - \frac{RT}{n_2\mathfrak{F}} \Sigma\, \gamma_{i''} \ln a_{i''}\right) \qquad (3)$$

There are many cases, however, in which the convention expressed by the first rule or by equation (3) leads to erroneous results. Suppose the student is required to deduce E° for the reaction

$$2Cu^+ \rightleftharpoons Cu + Cu^{++} \qquad (4)$$

from a table of data containing the half-reactions of copper (*1*). These are the possibilities:

		E°	*Volt equivalents*
(II)	$Cu \rightleftharpoons Cu^{++} + 2e$	−0.345	−0.690
	$2(Cu \rightleftharpoons Cu^+ + e)$	−0.522	−1.044
	$2Cu^+ \rightleftharpoons Cu + Cu^{++}$	0.177	0.354
(III)	$2(Cu^+ \rightleftharpoons Cu^{++} + e)$	−0.168	−0.336
	$Cu \rightleftharpoons Cu^{++} + 2e$	−0.345	−0.690
	$2Cu^+ \rightleftharpoons Cu + Cu^{++}$	0.177[2]	0.354
(IV)	$Cu^+ \rightleftharpoons Cu^{++} + e$	−0.168	−0.168
	$Cu \rightleftharpoons Cu^+ + e$	−0.522	−0.522
	$2Cu^+ \rightleftharpoons Cu + Cu^{++}$	0.354[3]	0.354

The first rule applies in examples I and IV, not because e. m. f.'s are additive as such, but because the e. m. f.'s are directly proportional to the free energies, which are additive. In examples II and III, typical of those first recognized by R. Luther (*6*), both half-reactions involve a single element in different oxidation states. In such cases the number of electrons transferred in the half-reactions may differ from the number transferred in the complete reaction. The e. m. f.'s, no longer proportional to the free energies, may not be manipulated like free energies and the first rule cannot be applied.

The second rule states that when two half-reactions are combined to give another half-reaction the standard

[1] Present address: Department of Chemistry, Illinois Institute of Technology, Chicago 16, Illinois.

[2] Incorrect.

[3] Correct.

free-energy changes, rather than the standard electrode potentials, are combined algebraically; from the result the new standard e. m. f. is derived. In these cases the use of volt equivalents shortens the calculations considerably.

		Volt equivalents	$E°$
(V)	$Cr \rightleftharpoons Cr^{+++} + 3e$	2.13	0.71
	$Cr^{++} \rightleftharpoons Cr^{+++} + e$	0.41	0.41
	$Cr \rightleftharpoons Cr^{++} + 2e$	1.72	0.86

The second rule may be worded so as to include all possible combinations. It is thermodynamically exact and, applied to the preceding examples, it invariably gives the correct e. m. f.

It seems desirable, therefore, to devise a presentation of standard electrode potentials which will avoid the errors inherent in the first rule, which will be consistent with theory, and yet which can be taught in general chemistry without making the treatment more difficult or arbitrary than it is at present. It will be assumed that Faraday's laws of electrolysis have been presented before the standard electrode potential is introduced. The following treatment is similar to that of Eastman and Rollefson (7) but a fundamental modification is introduced in the rule of combination.

A standard electrode potential is an intensive property of a given half-cell, and as such is nonadditive—it is independent of the number of chemical equivalents or faradays involved. On the other hand, when standard electrode potentials are to be combined they can be regarded as quantities of free energy in units of 23,100 cal., since $E°$ is proportional to $\Delta F°$ if n is taken as unity. Therefore, a table of standard electrode potentials is also a series of numbers in volt equivalents, characteristic for a half-reaction in which one chemical equivalent is involved. It is convenient to write the chemical half-reactions as in the following table so as to associate a one-faraday change with the standard electrode potential; whenever these half-reactions are combined the standard electrode potentials should be termed "volt equivalents" and treated additively.

Half-reaction	$E°$, *volts*
$\frac{1}{2}(Mg \rightleftharpoons Mg^{++} + 2e)$	2.34
$\frac{1}{2}(Cr \rightleftharpoons Cr^{++} + 2e)$	0.86
$\frac{1}{3}(Cr \rightleftharpoons Cr^{+++} + 3e)$	0.71
$Cr^{++} \rightleftharpoons Cr^{+++} + e$	0.41
$Cu^{+} \rightleftharpoons Cu^{++} + e$	−0.168
$\frac{1}{2}(Cu \rightleftharpoons Cu^{++} + 2e)$	−0.345
$Cu \rightleftharpoons Cu^{+} + e$	−0.522
$MnO_4^{--} \rightleftharpoons MnO_4^{-} + e$	−0.54
$\frac{1}{3}(4OH^{-} + MnO_2 \rightleftharpoons MnO_4^{--} + 2H_2O + 2e)$	−0.58
$\frac{1}{3}(Au \rightleftharpoons Au^{+++} + 3e)$	−1.42

To a first-year student the distinction between the volt and the volt equivalent need not be confusing. He has already learned to differentiate between some of the following related properties: weight and density, mols and concentration, force and pressure, heat capacity and specific heat, the speed of a reaction and its specific rate constant, in short, between extensive and intensive properties. He may also know how to convert the extensive into the intensive property by specifying a unit quantity of substance concerned.

If the student has understood the difference between weight and density he should not find it unnatural to characterize a cell by its standard e. m. f. in volts and work with another unit, the volt equivalent, when he is combining one half-cell with another. He is now prepared for a general rule applicable to all combinations of half-cells. *Manipulate both the chemical reaction and the volt equivalents in analogous algebraic operations until the resultant volt equivalents are associated with a change of one chemical equivalent in the combined reaction.* This number of volt equivalents can now be regarded as the cell or half-cell potential.

Several of the preceding examples are worked by this method to obtain standard e. m. f. values.

		Volt equivalents
	$\frac{1}{2}(Mg \rightleftharpoons Mg^{++} + 2e)$	2.34
(VI)	$\frac{1}{3}(Au \rightleftharpoons Au^{+++} + 3e)$	−1.42
	$\frac{1}{2}Mg + \frac{1}{3}Au^{+++} \rightleftharpoons \frac{1}{2}Mg^{++} + \frac{1}{3}Au$	3.76
	$E° = 3.76$ volts	

(VII)	$\frac{1}{2}(Cu \rightleftharpoons Cu^{++} + 2e)$	−0.345
	$Cu \rightleftharpoons Cu^{+} + e$	−0.522
	$Cu^{+} \rightleftharpoons \frac{1}{2}Cu + \frac{1}{2}Cu^{++}$	0.177
	$Cu^{+} + Cu^{+} \rightleftharpoons Cu + Cu^{++}$	0.354
	$E° = 0.354$ volt	

Notice that the first answer obtained in VII from addition must be doubled to give a reaction in which *only one electron is transferred.*

		Volt equivalents
(VIII)	$\frac{3}{2} \times \frac{1}{3}(Cr \rightleftharpoons Cr^{+++} + 3e)$	$\frac{3}{2} \times 0.71$
	$\frac{1}{3}(Cr^{++} \rightleftharpoons Cr^{+++} + e)$	$\frac{1}{2} \times 0.41$
	$\frac{1}{2}(Cr \rightleftharpoons Cr^{++} + 2e)$	0.86
	$E° = 0.86$ volt	

Finally the combination of a half-cell with a complete cell reaction is also accessible by this method.

		Volt equivalents
(IX)	$\frac{1}{3}(3MnO_4^{--} + 2H_2O \rightleftharpoons MnO_2 + 2MnO_4^{-} + 4OH^{-})$	0.04
	$\frac{1}{3}(4OH^{-} + MnO_2 \rightleftharpoons MnO_4^{--} + 2H_2O + 2e)$	−0.58
	$MnO_4^{--} \rightleftharpoons MnO_4^{-} + e$	−0.54
	$E° = 0.54$ volt	

If the half-reactions are written as they appear in the table, the rule of combination can be reformulated for more advanced students as

$$nE_{1,2} = E_1° - E_2° - \frac{RT}{\mathfrak{F}}\left(\sum \gamma_i' \ln a_i' - \sum \gamma_i'' \ln a_i''\right) \quad (5)$$

where n represents the number of electrons transferred in the complete reaction. In many cases $n = 1$, but when $n \neq 1$ the calculation requires an extra step, as in example VII.

With this rule of combination even the first-year student can use e. m. f. values to predict whether or not any reaction will go, and to make up new half-reactions.

Isolated facts in the chemistry of an element may take on new significance; for example, the disproportionation of the cuprous ion in aqueous solution and the requirements for stable cuprous compounds can be discussed in the light of the equilibria given in example VII. It is emphasized that, if electrode potentials are to be presented, the operations involved in the volt-equivalent method of combining half-reactions are no more arbitrary or difficult than those associated with the first rule. Once learned it can easily be applied in topics in general and analytical chemistry. Finally, the volt-equivalent method is thermodynamically sound so that the student will have nothing to unlearn later.

ACKNOWLEDGMENT

The author would like to express his appreciation to Professor Robert W. Parry for several helpful discussions.

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A "DERIVATION" OF THE NERNST EQUATION FOR ELEMENTARY QUANTITATIVE ANALYSIS

LOUIS MEITES
Yale University, New Haven, Connecticut

SOPHOMORES and juniors taking elementary quantitative analysis without previous instruction in physical chemistry frequently find their attempts to understand and apply the Nernst equation hampered by its presentation to them as a *fait accompli*. Knowing nothing of the considerations entering into its derivation, they are forced to use it in equilibrium constant and similar calculations without comprehending the fundamental relationship between the standard potential of a half-reaction and the equilibrium constant of a completed equation. Consequently one all too often finds that even the students who can perform the algebraic manipulations successfully do so as though performing a well memorized series of arbitrary mathematical steps.

This state of affairs is the logical result of the fact that a rigorous derivation of the Nernst equation is beyond the scope of such a course. The writer has for several years been experimenting with a "derivation" which, although clearly beneath criticism on thermodynamic grounds, produces the Nernst equation in a manner which the student can easily appreciate. Having found that their understanding of the applications of the equation is thereby greatly enhanced, he is impelled to present it here for the information of others troubled by the considerations outlined above.

Consider the half-reaction connecting ferrous and ferric ions:

$$Fe^{++} = Fe^{+++} + e \qquad (1)$$

The corresponding equilibrium constant would be

$$K = \frac{[Fe^{+++}]\,[e]}{[Fe^{++}]} \qquad (2)$$

Having already learned that acid-base reactions consist essentially of the transfer of protons, while redox reactions involve the transfer of electrons, the student is inclined to accept a parallel between the significance of the proton concentration in acid-base reactions and that of the "electron concentration" in redox reactions. Since in acid-base reaction theory one is concerned with the logarithm of the hydrogen ion concentration, the analogy can be pursued further by solving equation (2) for the logarithm of the "electron concentration." This gives

$$\log [e] = \log K + \log \frac{[Fe^{++}]}{[Fe^{+++}]} \qquad (3)$$

The next step is purely a matter of convention:

$$\log [e] = \log K - \log \frac{[Fe^{+++}]}{[Fe^{++}]} \qquad (4)$$

At about this point it is necessary to discuss the mean-

ing of the "electron concentration." Of course an aqueous solution containing ferrous and ferric ions contains no free electrons. (The difference between metallic and electrolytic conductors may be reviewed here.) So the "electron concentration" is not a physically significant quantity. It is merely a measure of the tendency of the solution to take up or give off electrons. If a solution contains a high concentration of ferric ion and a low concentration of ferrous ion, equation (1) would suggest the qualitative conclusion that reduction be much easier, and oxidation much more difficult, than if the relative concentrations were reversed. This is the same conclusion that would be reached on the basis of assuming a low "electron concentration" in the first solution and a much higher one in the second. Thus, although the "electron concentration" is fictitious, its use as an aid in reasoning leads to conclusions which are qualitatively correct.

Now, if this "electron concentration" is a measure of the tendency of the solution to react with or give up electrons, it should be possible to measure the difference between this tendency of one solution and the corresponding tendency for another solution. Let us arbitrarily assume that a solution containing 1 M hydrogen ion in equilibrium with hydrogen gas at one atmosphere pressure behaves as though it contained one mol of electrons per liter. (This is equivalent to defining the standard potential of the hydrogen: hydrogen-ion couple as zero. The instructor may now wish to digress to explain the difficulties involved in defining a single electrode potential, and to stress the fact that the final comparison between two solutions cannot depend on the standard to which the properties of both are referred.)

If an inert metal wire is immersed in this solution, it might be expected to assume the same charge density (*i. e.*, the same potential) as the solution itself. If another similar wire is immersed in a second solution, it would be expected to assume the potential of that solution. Now if the two wires are connected to the terminals of a high-resistance voltmeter, so that no current can flow to alter the compositions of the solutions during the measurement, the difference in potential can easily be measured. It may be pointed out that a difference in potential means that a redox reaction would occur if the two solutions were mixed, and that a large potential difference indicates that the reaction would be more nearly complete than if the potential difference were smaller.

Suppose that such a measurement is made of the potential difference between our standard hydrogen: hydrogen-ion solution and another hydrogen: hydrogen-ion solution containing, say, 10 M hydrogen ion. By comparison with equation (2) it will be found that the second solution will behave as though it contained ten mols of electrons per liter. The values of log $[e]$ for the two solutions thus differ by one unit. The voltmeter reading will be found to be 0.05915 volt at 25°. Then we can write

$$E = 0.05915 \log [e] \tag{5}$$

which when combined with equation (4), gives

$$E = 0.05915 \log K - 0.05915 \log \frac{[Fe^{+++}]}{[Fe^{++}]} \tag{6}$$

In a solution 1 M in both Fe^{++} and Fe^{+++} the second term on the right of equation (6) vanishes. The potential is then the potential under a sort of "standard condition" like the standard conditions involved in gas law calculations, and is called the "standard potential," $E°$. Since by equation (6) this is equal to 0.05915 log K, it follows that

$$E = E° - 0.05915 \log \frac{[Fe^{+++}]}{[Fe^{++}]} \tag{7}$$

By an entirely analogous procedure with, say, the manganous ion: permanganate couple, it is possible to show the significance of the value of n, and thus complete the derivation.

Even the common sign convention can be justified to the student's satisfaction. Consider a solution containing permanganate, manganous, and hydrogen ions, all at 1 M concentration: It is a strongly oxidizing solution, which means that it has a great affinity for electrons, or that it appears to have a very low "electron concentration." By equation (5), therefore, E (which in this case is equal to $E°$) is negative. Similarly $E°$ would be expected to be positive for, say, the chromous: chromic couple.

In calculating the equilibrium constant of a redox reaction one can of course set the potentials of the two couples equal at equilibrium and proceed in the conventional fashion. The following method, however, seems to be more readily appreciated by the student.

Taking the ferrous: permanganate reaction as an example:

$$E°_{Fe} = 0.05915 \log \frac{[Fe^{+++}]\,[e]}{[Fe^{++}]} \tag{8}$$

and

$$E°_{Mn} = \frac{0.05915}{5} \log \frac{[MnO_4^-]\,[H^+]^8\,[e]^5}{[Mn^{++}]} \tag{9}$$

Rewriting (8) as

$$E°_{Fe} = \frac{0.05915}{5} \log \frac{[Fe^{+++}]^5\,[e]^5}{[Fe^{++}]^5} \tag{10}$$

and subtracting (9) from (10) gives, after a little simple algebra,

$$E°_{Fe} - E°_{Mn} = \frac{0.05915}{5} \log K \tag{11}$$

The theoretical shortcomings of this presentation are so numerous and readily apparent that it seems unnecessary to list or discuss them. Nevertheless, the writer feels that its use is more than justified by the students' learning immediately that the Nernst equation is fundamentally nothing more than a means of describing equilibrium relationships in redox systems, and by their greater confidence in its use.

INTRODUCTORY EXPERIMENTS IN INORGANIC PAPER CHROMATOGRAPHY

JOHN G. SURAK and DONALD P. SCHLUETER
Marquette University, Milwaukee, Wisconsin

Although Tswett is usually given credit for originating the chromatographic method of analysis, his work was preceded almost 35 years by that of Schoenbein. While investigating the conditions of ozone formation under the influence of electrical discharges, he observed selective adsorption of the components of a mixture by the different heights to which they rose when a strip of filter paper was dipped in the solution. He called this new phenomenon "capillary analysis" and predicted its usefulness in the field of analytical chemistry (*1*). This was the inception of paper chromatography.

In 1944, after over 80 years of quiescence in this field, Gordon, Martin, and Synge extended Schoenbein's technique to two-dimensional chromatography (*2*). Their adaption was based on the partition principle which depends on a dynamic partition or distribution of dissolved or dispersed substance between two immiscible phases, one of which is moving past the other. The separability of mixtures depends primarily upon differences among the partition or distribution ratios of the solutes in the nonmobile and mobile phases.

Following the original work of Gordon, Martin, and Synge, a great number of variations in technique and apparatus have been published. The most widely used method employs strips of filter paper which are suspended from a trough containing water, a partially miscible organic solvent and usually a small amount of acid. A drop or streak of the solution to be analyzed is placed near the top of the strip and the paper and trough are placed in a closed container. After a suitable length of time, depending on the filter paper and solvent used, the strip is removed from the container and developed with reagents which will locate and identify the various constituents of the mixture. A very large number of both organic and inorganic substances have been separated by this method (*1*).

Since the field of chromatography is rapidly developing to encompass all phases of industry, it was felt that there was a need to develop a simple, small-scale method of paper chromatography which could be used in freshman qualitative-analysis classes. The majority of apparatus previously reported was not suitable for class participation (*5*). In the following procedure, the elementary chemistry student can be introduced to this rapid and convenient method of chemical analysis with a minimum of cost and effort.

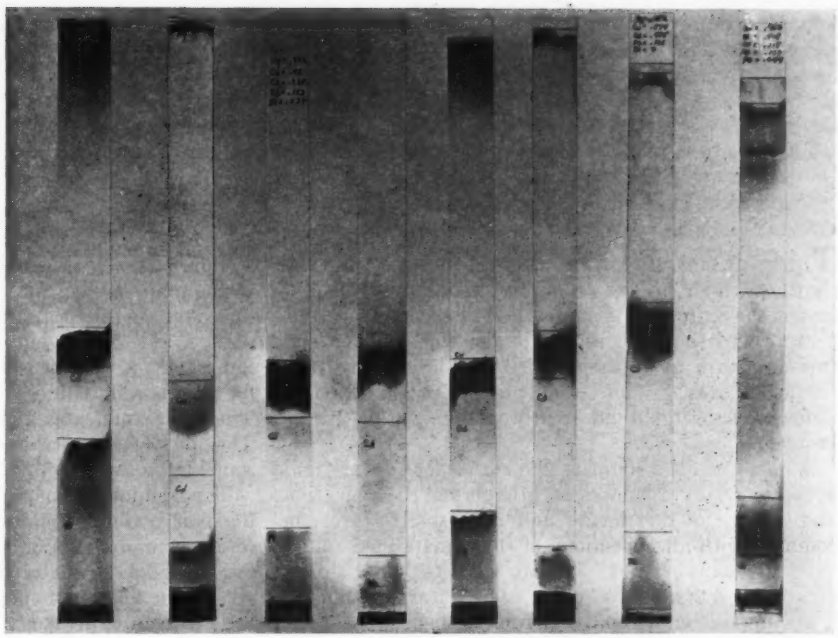
The equipment consists of a 300 × 29-mm. test tube and a loop of thin glass rod, the ends of which are inserted in a cork stopper. This glass loop prevents the paper strips from touching each other, since two strips are run simultaneously in one or more tubes. (A 300-ml. Erlenmeyer flask is used as a support for the tube.) For a large number of samples, a test-tube rack is made by drilling $1^1/_4$- inch holes in one side of a box, 36 × 6 × 6 in. About 33 holes are drilled. This is sufficient to hold one class's test tubes. The filter paper strips, 35 cm. long and 1.5 cm. wide, have a piece of glass rod inserted at the end to prevent curling of the paper. The procedure involves depositing a small amount of the sample mixture of ions on the paper by means of a fine capillary and allowing it to dry. After drying, the strip is placed in the test tube containing 10 ml. of developing solvent. This solvent may be a mixture of water, butanol, or tertiary butyl alcohol, and acetic or hydrochloric acid. The tube is then closed loosely with a cork until the solvent has reached the top of the test tube. The chromatogram is then removed and the separated cations are identified with suitable reagents. When dry, the strips are sprayed with liquid plastic to prevent fading of the identifying colors.

In the classroom application of this procedure the students are supplied with filter paper cut to the proper size, and the necessary constituents for their solvent mixture. Unknown mixtures ordinarily issued to the student for analysis by conventional methods are used. It must be pointed out at this point that there are two factors which must be carefully avoided when this method is introduced on a class basis. The air must be relatively free of hydrogen sulfide gas or precipitation of the unknown mixture will occur immediately after the sample solution is placed on the paper. In addition, the sample spot must be dry before the strip is placed in the tube or it will tend to be diluted by the developing solvent and a very poor separation will result. Of the many separations attempted the majority of failures could be attributed to one or both of these factors.

Migration of the developing solvent requires approximately 20 hours, after which time the strips are removed from the tube and allowed to dry. Group I cations are identified by spraying with potassium iodide or dichromate solution, while Groups II and IIA are identified by spraying the strips with distilled water and exposing them to hydrogen sulfide gas in a closed container for about 15 minutes. In order to simplify identification of the various cations several chromatograms are prepared for use as standards. A few strips

are run containing the entire group of cations which might be present in the student's unknown solution. By comparing the colors of the sulfides or iodides formed, and the extent of migration of his own strip with that of the standard chromatogram, the student is readily able to interpret his results. This system proved very satisfactory when introduced to a freshman chemistry class which had no previous knowledge of the chromatographic method.

A large number of developing solvents and grades of filter paper were investigated before a combination which would work under almost all conditions was obtained. A complete discussion of these experiments including investigation of various acid and pH ranges will be found in a subsequent paper. Only those solvents and papers used in the class experiments will be discussed here.



Chromatographic Separations Obtained by Students

The most satisfactory solvent mixtures consist of 15 per cent water, 10 per cent acetoacetic ester, 75 per cent butanol or tertiary butyl alcohol, and sufficient glacial acetic acid to adjust the pH between 3.5 and 4.0. Hydrion pH paper is used to measure the pH of the solutions. The acid is added to give more concise bands and prevent trailing due to hydrolysis and the existence of ions in complex and simple form at the same time (*4*). The acetoacetic ester functions in the same manner (*3*).

Two grades of filter paper yielded separations which differed chiefly in the speed of migration rather than in the degree of separation. Whatman filter paper Nos. 3 and 4 gave good separations, but the R_f values for the chromatograms using the No. 4 grade were slightly higher than for the No. 3 grade. The difference is in the porosity of the papers.

Several examples of separations obtained by the students are shown in the figure. The lighter bands are not too distinct in the photograph but their uppermost boundaries are indicated by a pencil line. It will be noted that the rate of migration of the various cations is strongly influenced by the other ions which are present in the sample solution.

Although this is a very simple and rapid method of qualitative analysis, it is not intended to replace the conventional method now employed. The fundamental purpose of this small scale procedure is to introduce this important analytical tool, chromatography, to the future chemist.

Work is under way on the remaining groups and it seems very likely that this same procedure will prove successful. A laboratory manual for the chromatographic separation of the cations and certain of the anions found in the conventional qualitative inorganic analysis course is in the process of preparation.

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A SOLUTION TO THE PROBLEM OF STORAGE AND AVAILABILITY OF CHEMICAL LITERATURE

THEODORE J. WILLIAMS and ARTHUR ROSE
The Pennsylvania State College, State College, Pennsylvania

THE great bulk of existing chemical literature and the continued high rate of production have greatly accelerated the search for some method or device to permit a rapid and thorough search of the whole breadth of this literature to verify the presence or absence of a particular idea or fact. The present paper describes a device that should aid greatly in the solution of this problem.

A microcard is a compact and convenient device, and is commercially available at present for the reduction and storage of documents and papers. However, in common with all collections of documents filed under "a single main heading" system, the location of subordinate subjects within a "deck" of microcards is difficult if not impossible without a complete inspection of the contents of the cards, or an elaborate cross-index system. If the microcard and its cross-index are combined with a coding and sorting system, it becomes possible to store very large quantities of information, and to sort out from this the cards containing the desired information in its complete form. Presently available IBM sorting machines can scan at the rate of 450 cards per minute and separate out all those cards corresponding to a previously specified number group or "code." IBM cards, combined with microcards, are therefore the proposal of the present paper.

DESCRIPTION OF CARD

A standard 3 × 5-in. microcard is of such a size as to fit neatly into a field comprising 60 columns of the standard 80-column IBM card. The remaining twenty columns are then available for coding purposes such as the following: (1) Ten columns are punched with a standard Dewey-Decimal or other similar classification of the main subject matter or title of the paper reproduced on the microcard. (2) Ten columns are punched with a set of randomly spaced, four-digit random numbers. A specific spacing and number is reserved for some specific subject heading designated by the user. The figure indicates the allocation of the card area. The proportions assigned to microprint and IBM punching may, of course, be modified if desired.

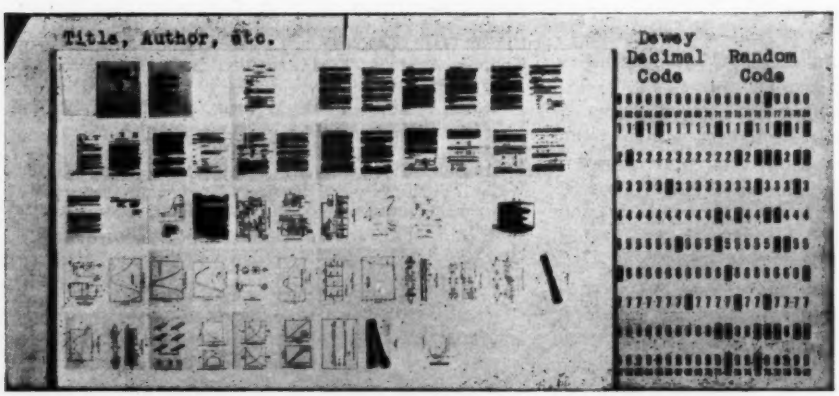
The randomly spaced four-digit random numbers make it possible to obtain up to several million different combinations of spacing and numbers useful for indexing. Also, the random spacing, both among and within the columns, makes possible the placing of the codes for up to 15 index headings on one card without noticeable weakening of the card. If additional headings are desired, they must be placed in successive duplicate micro-IBM cards.

Microcards must of necessity be reproduced by a photographic rather than a printing process. The photographic process nearly always causes curling or wrinkling of the photographic paper so that it will not pass through an IBM machine. However, the use of a plastic material such as the photographic film commonly used for film positives, if of the correct thickness, gives very satisfactory results. The required thickness for passage through IBM machines without special adjustments is from 0.006 to 0.008 in.

The best material of this type from the standpoint of readability is this same film made opaque by the inclusion of a white filler in the plastic, but ordinary film is also quite satisfactory.

The resulting plastic cards have shown themselves to be stronger and more durable than cards from standard IBM card stock. Several test samples of cards cut from film stock were passed through an IBM 080 Sorter over 50 times without visible wear while accompanying standard IBM cards were showing definite wear.

The plastic cards are more expensive than those of paper stock, but not appreciably more so than standard microcards, since the larger share of the cost of either



An IBM Microcard

type of microcard is the cost of the photographic process.

Some microcard readers will not take cards the size of standard IBM cards. However, models are available (for example, the "Readex") which will take material up to 6 × 9 in. in size.

THE CODING SYSTEM

As an example of the use of the random-digit-randomly-spaced method of coding, let us assume we have available the codes listed in Table 1.

TABLE 1
Sample Random Codes

	Columns									
Code	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
1			4		2		1		8	
2	8					0		1	3	
3	1		2			2				6
4	4		2	1			8			
5		9			8		4		2	
6				1	8	7	5			
7		6			9			5		2
8						4	2	1		8
9	5		7		3		1			
10		8				8			8	1

A study of this table will indicate the extreme variety of possibilities of codes available with this system. There are, of course, 10,000 possible four-digit numbers including zeros, and each number can be spaced in over 270 different ways. Table 1 shows three possibilities for the number 4218 in codes 1, 4, and 8.

If each of the above codes were assigned in such a way that they were all used in indexing some particular document, the punching in the random code field of the card would be as shown in Table 2.

TABLE 2
Example of Use of Random Codes

Column	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
Punchings	1	6	2	1	2	0	1	1	2	1
	4	8	4		3	2	2	5	3	2
	5	9	7		8	4	4		8	6
	8				9	7	5			8
						8	8			

In this case, 34 punchings are required to represent the ten four-digit numbers. Of course, combinations of digits and spacing could be developed to require a considerably fewer number of punchings if such were the object. The figure shows a card punched with the above ten random-digit codes and an accompanying Dewey-Decimal code.

It is, of course, obvious that the use of such a code as described above will necessitate a more bulky index of code numbers versus subject headings for use of the literature searcher than would a consecutively numbered system, since the card column location as well as value must be specified for each digit of the code. However, the random-digit coding system has several advantages which outweigh the above objection. They are:

(1) The random spacing of the punches in the card field permits the inclusion of a considerably greater number of index headings than the normal consecutively numbered code for a given permitted weakening of the card by means of the punchings.

(2) A random-digit code is by its very nature free of the usual normal fate of a consecutively numbered listing—the problem of including items omitted in the original coding which fall between consecutively numbered items on the preliminary list.

(3) Less sorting is required if a single column sorter is used since only a maximum of four columns need be checked for the random digits to locate any particular number, while for the consecutively numbered code a number of sorts equivalent to the number of decades in the largest number used is necessary. If a multiple column sorter such as the IBM Model 101 Statistical Machine, or the Model 080 equipped with a multiple column selector, is used, this advantage is of course of no consequence.

SORTING

When an 080 IBM Sorter is used, it is necessary, as mentioned previously, to scan the cards four times to sort out those having a particular random code number. Thus, if it is desired to obtain from a large deck those cards having code number 1 of Table 1, *i. e.*, the digits 4, 2, 1, 8 in columns 3, 5, 7, and 9, of the random digit field, the 080 sorter is first set to sort on column 3; thus separating those cards with a 4 punch in this column from all other cards. This smaller deck of cards is then sorted on column 5 to separate those cards containing a 2 punch. Successive sorting on columns 7 and 9 to separate the digits 1 and 8 completes the separation of the desired cards.

When using a sorter with multiple-column sorting characteristics, such as the Model 101 Statistical Machine, or the 080 Sorter equipped with a multiple column selector, the complete sorting operation can be done in one machine pass. Both of these machines are equipped with a control board whose use permits selection of a particular digit in each of several different card columns at once. Thus, one would wire the control board to select cards with the digits 4 in column 3, 2 in column 5, 1 in column 7, and 8 in column 9. Then as the cards are passed through the machine, those containing the desired combination of punches pass into one machine pocket while all others pass into another. The separation is thus completed in a single machine pass.

ACID-BASE TITRATION CURVES

An Experiment in Acidimetry

JAMES M. HENDEL
Hunter College, New York, New York

In the titration of an acid by standard alkali the graph showing how the pH of the acid solution changes with the addition of base may be used to locate the end point, which is taken as the mid-point of the portion of the curve parallel to the pH axis, or the point where the slope of the curve is maximum. The purpose of making such a graph is, of course, to guide one in the selection of the proper indicator for the particular acid titration. The method of determining the pH is usually electrometric, originally the hydrogen electrode, or latterly the glass electrode, being used in conjunction with a standard calomel electrode. The graphical results of such experiments may be obtained by a much simpler method requiring no experience in electrical measurements, which therefore could be introduced early in the course in quantitative analysis. This method involves the use of a mixed indicator solution, sometimes called a "universal" indicator. The curves obtained by the two methods are similar in form and practically coincide in the critical range between pH 5 to pH 9. The indicator method proves to be just as valid as the electrometric in judging which individual indicators are suitable for the titration in question.

The universal indicator solution[1] used in these experiments consisted of:

Methyl orange	0.05 g.
Methyl red	0.15 g.
Bromthymol blue	0.30 g.
Phenolphthalein	0.35 g.
66% Ethyl alcohol	1 liter

The colors exhibited by this indicator are:

Red	at pH 3 or less
Red orange	pH 4
Orange	pH 5
Yellow	pH 6
Yellow-green	pH 7
Green-blue	pH 8
Blue	pH 9
Violet	pH 10
Red-violet	pH 11 or more

The procedure in general was to take 25 ml. of approximately 0.1 *N* acid, add 1 ml. of universal indicator and 75 ml. of distilled water, and compare the color with that of a buffer solution of known pH containing the same proportion of indicator. Sufficient 0.1 *N* sodium hydroxide was then added to bring the color up to that of the next unit pH value and the volume of added base plotted against this value. Successive portions of base were then added to produce the colors shown by the buffers of known pH value, proceeding cautiously between pH 5 and pH 9 where a drop or two of base often was sufficient to raise the pH by one whole unit.

The buffer solutions used to produce the colors for comparison with the unknown solution were mixtures of 0.2 *M* Na_2HPO_4 with 0.1 *M* citric acid as follows:

pH	*Phosphate, ml.*	*Citric acid, ml.*
3	8.22	31.78
4	15.42	24.58
5	20.60	19.40
6	25.26	14.74
7	32.94	7.06
8	38.90	1.10

To each of these was added 0.40 ml. of universal indicator. To prepare the buffer for pH 9, 50 ml. was taken of a solution which was 0.2 *M* with respect both to H_3BO_3 and KCl, to which was added 21.40 ml. of 0.2 *M* NaOH, the mixture being then diluted to a volume of 200 ml.; for pH 10, 50 ml. of the boric acid-potassium chloride mixture was added to 43.90 ml. of 0.2 *M* NaOH, and diluted to 200 ml. To these two solutions, 2 ml. of universal indicator was added.

Preliminary experiments showed that the titration curves for hydrochloric and for acetic acid resembled in form the typical curves for a strong acid and weak acid as determined by electrometric methods. To show their identity the course of the colorimetric titration was followed with glass and saturated calomel electrodes. The tables and graphs presented here show the results obtained with the two indicator systems for acetic, succinic, and citric acids, where only one inflection point is obtained, and for phosphoric acid where two inflection points are to be expected. The reverse titration, of sodium carbonate by standard HCl, likewise showed close coincidence in the values by the two methods.

Values in Table 1 from a pH of 5 to 11 as found by the indicator method, are plotted in Figure 1. This plainly shows an inflection point at a pH of 7 and the practical identity of the values by the two methods, since the electrometric measurements shown as squares on the graph were reliable only to one-tenth of a pH unit.

The more important values in Table 2 are plotted in Figure 2. The maximum slope of this curve occurs at 24.85 ml. and a pH of 9. A titration using phenolphthalein required 24.90 ml. of base. But any other

[1] Smith, T. B., "Analytical Processes," Edward Arnold & Co., London, 1929, p. 363.

TABLE 1

Titration of 25 ml. of 0.0832 *N* Acetic Acid by 0.1004 *N* NaOH

Ml of NaOH	*Colorimetric pH*	*Electrometric pH*
0	3	3.3
4.60	4	4.2
11.37	5	4.6
19.15	6	5.8
20.50	7	7.0
20.60	8	7.7
20.70	9	8.9
21.05	10	9.8
22.00	11	10.7

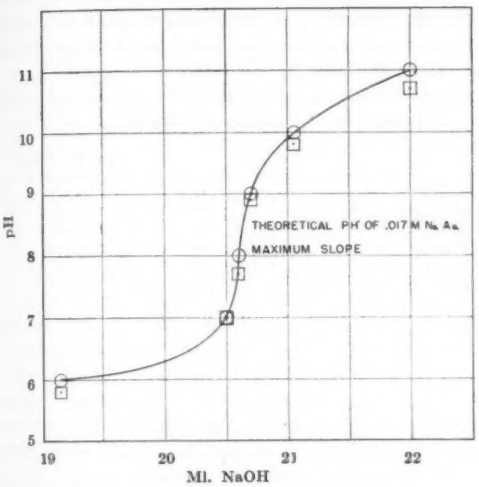


Figure 1. Acetic Acid Titration Curve
(Squares indicate electrometric values)

indicator changing within the range of a pH of 8 to 9.5 should prove useful.

In Table 3 the identity of the pH values by the two methods is obvious. The titration should require an indicator changing between pH 8 and 9. Two titrations using phenolphthalein used 30.85 ± .05 ml. of base showing the neutralization of all three hydrogen ions.

As a more rigorous test of the validity of the indicator method, titrations of phosphoric acid were carried out to see if the two inflection points corresponding to the neutralization of the first two hydrogen ions would appear. Table 4 and Figure 4 show the results.

TABLE 2

Titration of 25 ml. of 0.1000 *N* Succinic Acid by 0.1004 *N* NaOH

Ml of NaOH	*Colorimetric pH*	*Electrometric pH*
0	3	3.3
4.20	4	3.9
15.10	5	5.3
23.00	6	6.2
24.40	7	7.2
24.75	8	7.9
24.85	9	...
24.90	...	9.3
25.00	10	9.5
26.00	11	10.8

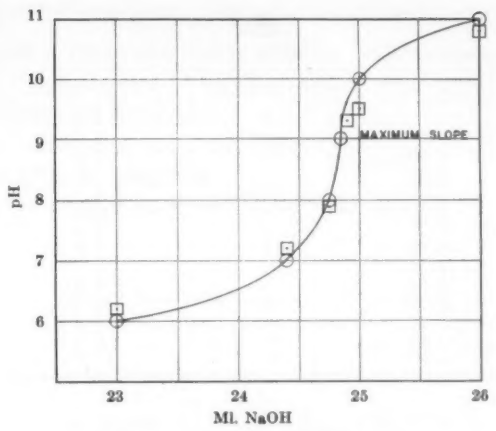


Figure 2. Succinic Acid Titration Curve
(Squares indicate electrometric values)

There are evidently two maxima in the curve, around pH 5 where 6.65 ml. of base were used for neutralization of the first hydrogen ion and near pH 10 corresponding to 13.45 ml. for the second hydrogen ion. A separate titration with phenolphthalein required 13.33 ml. of base.

A CLASS EXPERIMENT

With a view to introducing a little of the "research" attitude into the course in quantitative analysis and to illustrate the value of titration curves in deciding what indicator should be used for a particular acid-base

TABLE 3

Titration of 10 ml. of 0.300 *N* Citric Acid by 0.0973 *N* NaOH

Ml. of NaOH	*Colorimetric pH*	*Electrometric pH*
0	3	2.5
12.30	4	4.0
19.30	5	5.3
25.40	6	5.8
29.80	7	7.0
30.70	8	8.0
30.80	9	8.5
30.90	...	9.0
31.45	10	9.9

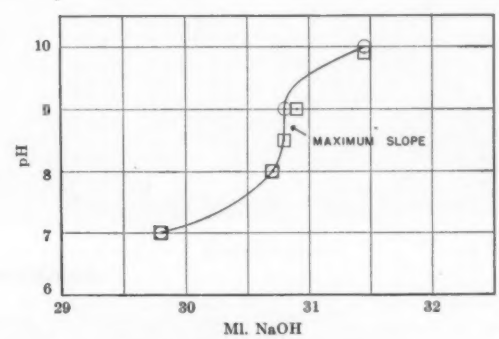


Figure 3. Citric Acid Titration Curve
(Squares indicate electrometric values)

TABLE 4
Titration of 25 ml. of 0.0259 *M* H_3PO_4 by 0.0973 *M* NaOH

Ml. of NaOH	*Colorimetric pH*	*Electrometric pH*
0	3	2.8
4.00	...	3.2
6.00	4	3.8
6.45	...	4.1
6.65	5	5.1
6.80	...	5.6
7.15	6	6.0
10.00	7	7.0
12.35	...	7.8
12.55	8	8.0
13.30	9	8.9
13.40	...	9.5
13.55	10	9.6
15.00	11	11.0

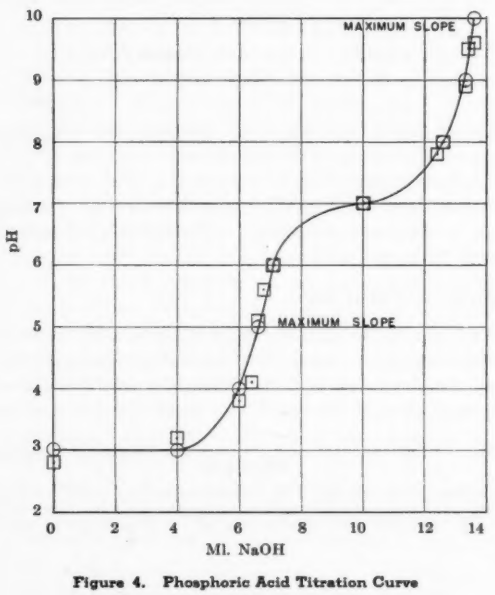


Figure 4. Phosphoric Acid Titration Curve
(Squares indicate electrometric values)

titration, each student was given a different acid and told to make 250 ml. of solution approximately 0.1 *N*. Since some of the acids were not soluble to that extent in cold water, these were dissolved in 95 per cent ethyl alcohol or in hot water to make a concentration requiring about 25 ml. of standard 0.1 *N* NaOH. In the usual case 25 ml. of the 0.1 *N* acid was diluted to 100 ml., 1 ml. of universal indicator was added, and the titration was carried out, adding sufficient base each time to change the pH by one unit as shown by the indicator. The comparison buffer solutions were provided by the instructor, made up in "wholesale" quantities. From the resulting titration curve the student was required to select three indicators which might be used to locate the end point, and to determine, by titrating, which one of these was the best. The indicators supplied were bromphenol blue, methyl red, bromcresol purple, bromthymol blue, phenol red, cresol red, thymolphthalein, and thymol blue. The use of methyl orange or phenolphthalein was discouraged since one of the minor objectives of the experiment was to de-emphasize these two indicators.

Curves showing unmistakable inflection points were obtained for the following acids:

Acid	K_a	*pH of vertical portion*
Trichloroacetic	2×10^{-1}	4–11
Dichloroacetic	5×10^{-2}	5–9
Salicylic	1.1×10^{-3}	6–9
Formic	1.8×10^{-4}	7–9
Lactic	1.4×10^{-4}	7–9
Benzoic	6.3×10^{-5}	8–9

The curves for phthalic acid, $K_1 = 1.3 \times 10^{-3}$, and tartaric acid, $K_1 = 1.1 \times 10^{-3}$, showed a vertical portion in the curve only after neutralization of the second hydrogen ion, pH 8–9 for the former and pH 7–9 for the latter. Boric acid, $K_1 = 6.4 \times 10^{-10}$, with 25 ml. of glycerol added to 25 ml. of 0.1000 *M* solution showed a maximum slope between pH 8–9 corresponding to 24.06 ml. and 24.60 ml. of 0.1043 *N* NaOH; the theoretical volume for the first hydrogen is 23.97 ml. of base. A titration with phenol red (7.0–7.4) required 23.83 ml., with cresol red (7.6–8.2) 24.02 ml. and with phenolphthalein 24.03 ml. of base.

For comparison with the experimental curve it is of value to plot the theoretical graph. For weak monobasic acids the ionization constant will yield the initial pH, the hydrolysis formula for the salt will give the pH at the stoichiometric point, and for intermediate points the equation

$$\mathrm{pH} = \mathrm{p}K_a + \log \frac{(\mathrm{salt})}{(\mathrm{acid})}$$

will serve the purpose. This correlation of experiment and theory arouses considerable interest on the part of the student; it is not just another recipe.

CHEMISTRY WORKSHOP

The third annual "workshop" for general chemistry will be held from June 11 to 21 at Oklahoma A & M College, Stillwater, Oklahoma. It is again under the auspices of the Division's Committee on the Teaching of Chemistry, of which Dr. O. M. Smith is chairman. Further details will be announced later.

RAYMOND DELABY

CLÉMENT DUVAL
University of Paris, Paris, France
RALPH E. OESPER
University of Cincinnati, Cincinnati, Ohio

RAYMOND (Marie Florent) Delaby well merits his international reputation for two reasons: his competent conduct of the general-secretaryship of the International Union of Chemistry, and his excellent research studies in the fields of organic, pharmaceutical, and analytical chemistry.

Born at Lens (Pas de Calais) on September 20, 1891, his scientific training was begun at the Faculty of Medicine and Pharmacy in Lille, where he was assistant 1912–13. The *pharmacien* degree was obtained in 1913. Shortly after the outbreak of World War I he volunteered for active duty. He was gassed at Verdun in 1916 and after his recovery was appointed *chef du laboratoire de toxicologie.* The Croix de Guerre was awarded in 1916. At present he holds the rank of *pharmacien commandant de reserve.*

After the war he continued his education at Paris and received his *docteur ès sciences physiques* at the Sorbonne in 1923. Having now resolved to enter the teaching profession, he qualified as *agrégé* (teaching fellow) at the Faculté de Pharmacie in 1926. In 1937 he was appointed professor of analytical chemistry in this school and in 1940 exchanged this chair for his present post, professor of pharmaceutical chemistry. He has been an outstanding teacher, both for elementary and advanced students. He worked out new spot-detection methods for arsenic, antimony, and tin, and in line with this interest with the collaboration of J. A. Gautier, he composed the well-known "Analyse qualitative minérale a l'aide des stilliréactions" (2nd ed. 1949).

Delaby did his doctorate work under Marcel Delépine,[1] who suggested as a thesis topic a study of the homologues of glycerol in the acyclic and heterocyclic series. The organo-magnesium compounds were condensed with acrolein, and the resulting vinyl alcoyl carbinols were then converted to the glycerols via the bromhydrins. Delaby prepared derivatives of these new compounds, and also used them in the classic Skraup synthesis to arrive at new quinolinic derivatives. The section "Triols et leurs derivés," in Grignard's "Traité de chimie organique" was logically entrusted to Delaby.

Selected examples from his many research projects will demonstrate the caliber of the work that has come from his laboratory. Straight chain α,β-ethylenic aldehydes were prepared through the isomerization of vinylaryl and vinylaracoyl carbinols into β-homoacroleins. With J. Lecomte he studied the infrared absorption spectra of about 60 ethylenic derivatives which he had prepared. He confirmed the existence of sarothamnine, a satellite alkaloid of spartein, whose constitution he had clarified.

He investigated the group sulfamides-amidines from the pharmacological viewpoint, and dealt with *p*-sulfamidobenzamide in particular. With R. Charonnat he prepared dioxypyramidone (α-methyl-α-acetyl-β-dimethyloxamyl-β-phenylhydrazine) and proved that though less toxic than pyramidone its analgesic properties are just as good.

In the field of organic analysis he and Y. Breugnot developed a new method for determining the acetyl index, namely acetylation in pyridine. The determination of streptomycin by means of periodic acid was worked out in his laboratory.

He, Charonnat, and M. Janot made an extended study of the radioactivity of the mineral waters of the eastern parts of France. Those coming from the Ballon d'-Alsace, the highest peak in the Vosges mountains, were found to be the most active. He has directed an excellent scientific film, "Vichy, capitale thermale."

Raymond Delaby

He has been a member of the Higher Council for Scientific Research since 1939. As *inspecteur genéral des établissements classés insalubres,* and also as *président du comité consultatif des établissements classés,*

[1] OESPER, R. E., J. CHEM. EDUC., 27, 567 (1950).

he devoted much of his time and energy to safeguarding the public health in Paris where the multiplicity of manufacturing establishments constitutes a serious problem.

Professor Delaby served as secretary-general of the Société chimique de France for 13 years. In 1947 he was unanimously elected to the presidency of this important organization. Since 1945 he has been secretary-general of the Fédération Nationale des Associations de Chimie en France. He has held the distinguished post of secretary-general of the International Union of Chemistry since 1945. After the close of the World War II, he again organized its various activities and had a prominent part in arranging the London (1949) and the New York (1951) meetings.

His services have been recognized by the award of many prizes, including the Lavoisier Medal. He is a member of the Académie de Médecine. In 1949 he was made *officier* of the Legion of Honor.

TECHNIQUES OF JOB HUNTING FOR THE CHEMIST

BENJAMIN N. NELSON
Westinghouse Electric Corporation, Pittsburgh, Pennsylvania

It is well known that jobs for chemists are becoming more plentiful in these days of industrial expansion. Nevertheless, it still takes work on your part to find the specific kind of job you want. You can lessen the problem by organizing the many specialized job-hunting techniques into a campaign made to suit your specific case. Whether you are a graduating student, an unemployed chemist, or one looking for a better job, you would want to find the answer to three questions: (1) What openings exist now or in the near future? (2) Which should I look into? and (3) How should I go about it?

WHAT OPENINGS EXIST?

Your best source of information on what openings exist now or may exist are the technical periodicals. *Chemical and Engineering News* is your best starting point, for not only does it advertise jobs available and wanted but it indicates future trends. The trends and activities of companies are revealed by studies of the editorial, news, financial, and advertising aspects of the magazine. In addition to that magazine there are others which have want ad columns and business information. There are general magazines like *Chemical Engineering, Chemical Industries, The Chemist,* and *Chemical Engineering Progress.* You will also find specialized magazines like *American Ceramic Society Bulletin, The Journal of American Oil Chemists Society, Soap and Sanitary Chemicals, American Perfumer and Essential Oil Review, Rubber Age, Modern Plastics, Metals Review, Food Industries, Instruments, Journal of the Electrochemical Society, American Dyestuff Reporter, Textile World,* and *The Paper and Trade Journal.*

In newspapers you will find classified advertisement listings of both private industry and agencies. From technical agency listings you can become acquainted with the trends and salary ranges in the various specialties. By studying business trends in the financial section you gain ideas of future needs. Furthermore, in the Sunday financial section of a large paper such as the *New York Times* you will find additional job openings listed, usually of an administrative and specialized type.

If you are interested in government employment, there are numerous agencies that can be contacted. Federal organizations such as the Bureau of Standards, Department of Agriculture, Department of Public Health, Air Force Cambridge Research Laboratories, Army Chemical Center, Naval Ordnance, and other ordnance laboratories employ many chemists. The Army and Navy maintain civilian agencies for scientific personnel at Washington, D. C., such as the Office of Naval Research and the Employment Coordinator of the Army. State, county, and city laboratories are another group within the civil service category. You can contact the laboratories directly or obtain civil service announcements and blanks at all important post offices. Remember, however, that it may take more than a year after application before an offer is

made. It is always worth while, however, to procure a civil service rating so that you have an idea of your worth according to their standards. You can also file applications at the Atomic Energy Commission, Washington, D. C., which will relay copies of the applications to its various installations.

Another important source of job openings is the Ameircan Chemical Society clearing house procedure. This has become an integral part of all national meetings and some local ones. Employees are personally interviewed by employers, which is an ideal situation for all concerned. You can also file applications by mail at clearing houses maintained at Chicago, New York, and Washington, where employers can see them and contact you.

Still another source of jobs lies in the use of government, private, or school agencies. Private agencies in large cities have good listings and are in contact with many employers. They can find jobs for you providing you are willing to give a large proportion of your first month's salary. Pick out technical types of agencies and file applications at their offices. They not only advertise available openings but circulate lists of available employees to prospective employers. Particulary if you are an unemployed chemist, the United States employment agency and school alumni office might be of considerable assistance to you.

There are various listings that supply information on laboratories that have potential openings. One excellent book is "Industrial Research Laboratories of the United States," 9th ed., 1950. This book classifies laboratories as to type of product manufactured, type of laboratory, location, number of chemists employed, and persons in charge. Other catalogues include "Chemical Industries Buyer's Guide," "Chemical Engineering Catalogue," business and industrial directories of the various states and cities, specialized industry catalogues, and McGraw-Hill special mailing lists.

WHICH OPENINGS SHOULD I LOOK INTO?

You now have before you lists of definite job openings and potential employers. The next problem is to decide which to approach first. You might consider type of organization, size, and location. Do you want to work for an industrial, government, or academic organization? If you are interested in an industrial company you might consider research, production, development, consulting, administrative, editorial, or sales types of positions. In government employment your work might be of a research, service, or administrative nature. If your interest is in an academic institution, you might consider teaching, graduate study, or research.

Of course, coupled with your interest in a type of work, should be your ability to do that work. Try to make an honest appraisal of what you are capable of doing to assure that you don't place yourself in a position where you hinder yourself in advancing. You may find it not too difficult to find the kind of positon you want, but to hold and advance in that job requires more than interest and ambition. In attempting to appraise your abilities you might find it helpful to consult your former teachers, supervisors, co-workers, friends, and vocational and personnel experts. Psychological testing of aptitudes as given at schools, the Veteran's Administration and by private concerns would yield much valuable information on the subject. Another good source of information is the reading of articles on the subject in *Chemical and Engineering News, The Chemist,* and JOURNAL OF CHEMICAL EDUCATION.

As to the size of the company, small and large organizations each have their merits. In the small company you may stand out more, advance more readily if you are good, and your duties may be of greater variety than in a large company. In the large company you may be in a more stable type of organization with large company benefits such as low-cost group insurance, good pension and hospitalization plans. Also, in the large company there may be more opportunity for specialization and research and the facilities and equipment may be more adequate than in a small company.

Concerning location, you might consider local industry or out-of-town industry. A position for local industry can be obtained more quickly since you can utilize techniques like the telephone and personal interviews. On the other hand, as an out-of-town seeker you would be screened first by mail correspondence.

You might also consider the advantages of a small town or large city. In the small town the cost of living may be relatively low and the environment healthier but the social, cultural, and educational facilities may be limited as compared to the large city.

CONTACTING THE EMPLOYER

Now that you know where to look for possible openings and what type of position you want, plan an efficient program of job seeking. As a student or employed chemist you would probably have more time available for investigating the possibilities than the unemployed chemist. The program then is different for each situation. But before going into this, a few hints are in order on three major job-hunting techniques: the writing of the advertisement, the letter, and the interview.

In writing your advertisement in a magazine, gain the interest of the employer by using a selling phrase to convey the thought that you can offer the employer something he needs. You can gain ideas by noting the wording of large company advertisements of job openings.

In writing a letter you must keep in mind that your success in being called for an interview will depend not only on what you have to offer but on how you present your information. In over-all appearance, for example, the letter should be in an easily opened standard envelope, neatly typed (or handwritten if quite legible) with sufficient paragraphs to break up the monotony of solid writing. Summaries of personal data, education,

and experience should be stated in an outline form, preferably on a separate sheet. In the contents of the first letter use an opening and closing that are not commonplace. Right from the beginning endeavor to make your letter stand out by stating your purpose in writing in a saleslike, honest manner. Give a complete summary of experience, a good photograph, and references in the first letter. Indicate that you have something to offer the employer by being specific in your statements of pertinent training and experience. In general you should avoid generalities, unessentials, apologies, monotony, and hackneyed statements. Don't seek sympathy from the employer or put him under any obligation. Remember, the purpose of your letter is to gain the employer's interest in you so that he will want to meet you and talk further.

If you are going to send letters to a large number of prospective employers it might be wise to consider some time savers. Of course, your letter to the most important employers should be composed to cater to his specific wants. However, for the balance of the prospective employers—those of secondary importance—considerable time can be saved by using copy methods. The important thing here is to have a good form letter to start with and to reproduce it so that it looks as if it was typewritten. You might consider Multigraph, Multilith, Hoover, Letter press, or Flatbed press methods. The latter three are the most similar to the typewritten characters and you can type in individual addresses if you have a ribbon similar to the one used on the machine. An expert can fill in addresses on any of these that will pass as a pure typewritten letter. Signatures can be made on a woodcut or a metal plate, although the former is sometimes more natural in appearance. The important thing to remember in this type of copy work is the advantage of time saving and extensive coverage of prospects—and this is especially useful in seeking positions of a general nature. However, if you are applying for a specialized type of work in a particularly desirable organization the extra time spent in designing an individual letter for the specific employer is by far the best method to use.

Assuming your letter has brought you an interview, you can prepare certain questions beforehand. If you know who is going to interview you, a knowledge of the man's special interests might be of benefit to you during your discussion. Sometimes you can gain such information from "American Men of Science" or other "who's who" type of book. You should also be acquainted with the company's products, activities, economic status, and competitive position. For sources of information use the periodicals and buyer's guide catalogues. By expressing interest in the company you can make a better impression on the interviewer. You too, however, can make good use of the interview by preparing beforehand a series of questions designed to reveal the nature of the job and the salary in terms of opportunity and risk of short-term employment.

With these techniques in mind let us consider first the specific program of the student. As a student, you will be made aware of various openings through school interviews with prospective employers, but you can go far beyond this. For government employment apply a year before graduation. For all types of positions you can gain much by attending the employment clearing house of American Chemical Society meetings. Several months before graduation put your advertisement in *Chemical and Engineering News*, file applications at the American Chemical Society clearing house centers, begin answering ads and writing letters. Keep in mind the hints on ads and letters given earlier in this article. If all these things are done, you will be able to make your decision in time for graduation.

As an unemployed chemist your most speedy methods are advertisement answering, making phone calls and paying personal visits to local companies, United States and private agencies, school alumni offices, and meeting clearing houses. By far the most rapid method of all is the personal visit to the company, seeing the right man, and filling out the necessary blanks while there. By looking through the book, "Industrial Research Laboratories of the United States," and some of the special lists available, you can sometimes find out who is the right man. When you see this man make good use of your time with him, keeping in mind the hints on interviews given earlier in this article. Remember, the interview is the most important step in getting the job. Since any other method used will ultimately end up as the personal interview, the earliest use of this technique is advisable.

If you are an employed chemist seeking a better position, you can usually take your time and do a more thorough job. All of the methods mentioned previously should be used but the stress is usually upon ads and letters. If you wish to keep your job hunting confidential, then interviews would have to be arranged evenings, on week ends, or on vacations. However, you might find it more advantageous to let your present supervisor know of your intentions. In that way you can take leaves of absence, deductions from vacations, and attend employment clearing houses at meetings. Your employer may be able to give you some very good ideas on possible openings. Furthermore, letting your employer know of your intentions is desirable when he is called upon to write a reference, which is usually the case. For thoroughness, you can undertake a six months' campaign of placing ads, answering ads, writing many letters, contacting people personally, and visiting the various types of agencies. By the end of that time you will know whether you can better yourself elsewhere.

In summary, if you make a careful and serious search for a job, taking the time to exploit each of the appropriate approaches, you will surely have some gratifying results. Consider it as a scientific project in which you are trying to find where your abilities, honestly appraised, can be utilized with greatest advantage. Your aim should be to find an organization where you can grow in professional status because of your advantage to the company.

Proceedings of the

PACIFIC SOUTHWEST ASSOCIATION OF CHEMISTRY TEACHERS

THIRD ANNUAL MEETING

The Pacific Southwest Association of Chemistry Teachers held its third annual meeting in San Francisco on November 2 and 3, 1951. The theme for the two-day period centered around "More effective science teaching in our colleges."

Dr. Howard Benninghoff, City College of San Francisco and President-elect of the Northern Section, acted as Program Chairman. The opening session was a dinner meeting at which Dr. Harry Redeker, President of the Northern Section, presided. Dr. N. E. Ballou of the Hunter's Point Radiation Laboratory spoke on "Recent developments in nuclear chemistry" and stressed material of this nature which could be incorporated into a college freshman or high-school chemistry course.

At the Saturday meeting, held at the City College of San Francisco, Dr. Redeker introduced the members of a panel who spoke on the topic, "Effective science teaching in our colleges." Members participating were: Dr. Howard Benninghoff, panel chairman; Dr. John Wise, Stanford University; Dr. Bentley Edwards, Chico State College, who presented the findings of the A. C. S. Oklahoma Workshop pertinent to this discussion; Dr. P. Victor Peterson, President of Long Beach State College, who represented the views held by the State Department of Education; Dr. Robert Stollberg, San Francisco State College; and Dr. L. Lykken, Shell Development Company.

Dr. Peterson, told of the "formula" which he used in making the final selection of a staff member. The prospective teacher must be basically well trained in chemistry and he must "like kids." Dr. John Wise followed and stressed the value of a good foundation in mathematics in grasping the basic fundamentals in college chemistry.

Dr. Stollberg announced that he wished to take the liberty of rewording the topic to read, "*Effect of* science teaching in the colleges." The high-school teacher, under many pressures, is confronted with a variety of aims in teaching chemistry: social relationships, clear and logical thinking, personality development, personal adjustment of the student, development of student initiative, facts and fundamentals of chemistry preparation for later specialization in science, or gearing the course to the majority of nonchemistry majors.

Dr. Lykken concluded the panel presentation by listing numerous characteristics in which industry is interested when selecting chemists.

In the general discussion, preparation in physics and mathematics was considered much more important than high-school chemistry.

The afternoon program, with Dr. Richard Wistar of Mills College and Association Treasurer presiding, consisted of the following papers.

Dr. John Wise, Stanford University, in his paper, "Teaching equilibrium constants in qualitative analysis," objected to the usual method of deriving the equilibrium constant expression from a discussion of the reaction rates of the reactants and the products because most reactions are not as simple as this derivation assumes. He recommended the presentation of the expression as a statement of the findings of experimental work. For second-year qualitative analysis he recommended the inclusion of work with equilibrium constants for oxidation-reduction reactions by the method outlined in Hogness and Johnson, "Chemical Equilibrium and Qualitative Analysis."

Dr. Norman Lofgren, Chico State College, presented a paper, "An experiment in the problem approach to teaching of college chemistry," in which he told of a course developed at Chico based upon the interest and initiative of the students who work on individual problems selected from each of five general topics. He illustrated his talk with slides taken from topics of the course, such as "Solutions." The chief difficulty encountered in presenting a course of this nature was its evaluation, but it was felt by the speaker that the students learned as much chemistry as in the regular course.

Mr. Rae Schwenk, Sacramento Junior College, choosing a "Salesmanship" topic, stressed the point that "we cannot sell chemistry unless we are sold on being chemistry teachers."

Mr. Raymond Martin, Sacramento Junior College, discussing "An effective method of teaching pH calculation," explained new teaching devices. The students first learn the logs to two places for three prime numbers 2, 3, and 7, and calculate others from these as needed. Students soon become able by this method to find pH by mental calculations.

Dr. Arthur Furst, University of San Francisco, described "A course in chemical literature." In this course, students are instructed in how to make the best use of library facilities, to find the most authentic sources on a given subject, to collect pertinent data, and to write the results in acceptable form for publication. Several students have had papers published, which were worked up in this course.

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

EFFECTS OF MOLECULAR SHAPES[1]

LAURENCE S. FOSTER
Watertown Arsenal Laboratory, Watertown, Massachusetts

For a long time the general course in freshman chemistry has been becoming more theoretical and less factual. No longer is it believed desirable to stuff students full of facts as a turkey is filled with stuffing. The student needs facts, but only enough to enable him to think about theories. He needs theories so that he does not need to know so many facts. If a student is asked "What are the probable valences of *astatine?*" he does not have to go to the laboratory to find out. He can make a reasonably good guess from the position of element 85 among the halogens in the periodic table. If he is asked "What are the valences of *tungsten?*" he may not be able to predict these so readily, but it is still conceivable that he will predict some of the valences of tungsten from his knowledge of chromium with reasonably good results. The periodic table thus permits a certain amount of deductive reasoning, and the student gradually learns what can be deduced from it.

Many physical and chemical properties of substances are, however, not dependent alone upon the location of the constituent elements in the periodic table. The shapes of the "molecules" are also very important. This is a neglected subject in general chemistry and should no longer be omitted.

A new book has just appeared by Walter Hückel—now available in English translation—with the title "Structural Chemistry of Inorganic Compounds."[2] The preface states, "This work attempts to furnish inorganic chemistry with that which organic chemistry has long possessed as a basis for its systematization, namely, a structural and constitutional theory in one embracing representation. This simultaneously points the way to a systematization of inorganic compounds, which, other than that based upon a classification according to the elements, is founded upon the properties that are governed by constitutional peculiarities.

"The perspective obtained thereby is quite different in nature from that which results when inorganic chemistry is treated element for element.... It should.... be clear that a correctly drawn up inorganic structural chemistry, taken in conjunction with the organic, provides a magnificent and internally consistent overall picture of the structure and constitution of matter."

To point out rather dramatically the difference that structure may introduce into molecules, consider the two simple and comparable substances, CO_2 and SiO_2.

Carbon and silicon are in the same periodic group: both have a valence of four in these compounds. Carbonates, with the $—CO_3$ radical are similar to metasilicates with the $—SiO_3$ group; yet how different the two simple substances CO_2 and SiO_2 appear to be! The differences are directly related to differences in structure. The existence of the carbonate radical, CO_3^{--}, shows that there is room for three oxygen atoms around the carbon atom. The SiO_3 metasilicate radical has a similar structure, but there is also the orthosilicate radical, SiO_4^{--}, that shows the silicon atom to be large enough to become surrounded by four oxygen atoms. No orthocarbonate is known. The carbon atom is too small to hold four oxygen atoms. A slight difference in size of the two atoms, silicon and carbon, is the only apparent difference between them. This, however, is enough to explain the difference between the two compounds, CO_2 and SiO_2. In solid silicon dioxide there is room enough to have each silicon atom bonded to four oxygen atoms in a tetrahedral arrangement and a giant molecule of SiO_2 with a melting point of 1700°C. forms. CO_2 cannot be bonded together similarly, so it forms only a weakly bonded solid that sublimes at −78°C. The problem is actually a little more complicated than this. The oxygen atoms in CO_2 are linear:

$$:\ddot{O}::C::\ddot{O}:$$

[1] Presented at the 13th Summer Conference of the New England Association of Chemistry Teachers, University of Rhode Island, Kingston, R. I., August 21, 1951, as part of a Symposium on the Content of the General Chemistry Course.

[2] Elsevier Publishing Co., New York, 1950. Translated by L. H. Long.

The SiO_2 molecule on the other hand, when formed into a giant molecule, no longer has a linear configuration, but consists of SiO_4 tetrahedra linked together so that every oxygen atom is common to two tetrahedra (thus retaining the SiO_2 composition).

The difference between CO_2 and SiO_2 is not directly a result of the electronic configuration but in spite of it. By rights, CO_2 should not have a linear configuration, but due to repulsion of the oxygen ions for each other a linear molecule results.

Another example shows again the importance of molecular shape. This example probably makes the difference between life on the planet Earth or the lack of it. The example is water, which has an extraordinarily high melting point and boiling point. Water is also unusual in that its ice is less dense than the liquid; ice floats on top of ponds instead of sinking to the bottom. Of all the simple hydrides of the subgroup B elements, water has by far the highest boiling point.[3] Instead of being a gas like methane, it boils nearly 300° higher than methane. Methane has a symmetrical shape:

Water might be thought also to have a symmetrical shape: H—O—H, but numerous laboratory experiments show that this cannot be so. The water molecule is bent with an angle of 105° between the two hydrogen-oxygen bonds.

Recently the location of the hydrogen atoms in ice has been settled by neutron bombardment experiments. Because of too feeble scattering by protons, electron diffraction and X-ray diffraction experiments do not reveal the hydrogen, but neutrons give their location easily.

Laboriously, over a long period of time, scientists have evolved ideas concerning the geometric shapes of inorganic structures. For example, the four molecules of ammonia around the copper ion in the complex ion, $[Cu(NH_3)_4]^{++}$, are at the corners of a square instead of being at the corners of a tetrahedron. The water molecules in $[Cu(H_2O)_4]^{++}$ are also at the corners of a square. The other water molecule in $CuSO_4 \cdot 5H_2O$ is attached to the SO_4 radical. With nickel the $4NH_3$ molecules can be either in a square or in a tetrahedron; when in a square the complex is red, and when in a tetrahedron, the color is blue-green.

It is seen, thus, that while the primary valence determines the stoichiometric formula, the properties depend also upon the structural relationships. In fact, structure may even determine which valence is exhibited.

[3] See the chart in PAULING, L., "General Chemistry," W. H. Freeman and Co., San Francisco, 1947, p. 271.

What is the explanation of these various structures? A good theory has been evolved based on the arrangement of the electrons around the nuclei of the various atoms. As you will see from a table of atomic structures, electrons are classified as *s*, *p*, *d*, and *f*.

The *p* orbitals evidently have a preferred orientation in space, along lines at right angles to each other, whereas *s* orbitals have no preferred orientation. With this basic information it is possible to explain the molecular shapes after both *s* and *p* electrons are used in making bonds, and a process called "hybridization" occurs. Let us illustrate this by an example.

The structure of oxygen is

$$\overset{s\ \ s\ \ p}{(8)\ 2)\ 2,\ 4}$$

There are two covalent bonds to an oxygen atom in water, and the oxygen atom makes use of two *p* orbitals to bind the hydrogen atoms. Since two *p* orbitals arrange themselves at right angles, the two bonds should be at right angles, but are 105° instead of 90°, because of the repulsion of the protons for each other. Hydrogen sulfide also has this structure, but the angle is 92°, the proton repulsion being less.

The CO_2 molecule uses all the valence electrons of the carbon atom

$$\overset{s\ \ s\ \ p}{(6)\ 2)\ 2,\ 2}$$

The result is a hybrid bond involving use of both *s* and *p* electrons —since the *s* orbital has no directional property, it points away from the *p* orbital and we get an angle of 180° between the bonds, that is a linear molecule.

When there are four covalent bonds they may point towards the corner of a square or towards those of a tetrahedron. The tetrahedral arrangement, as in methane, arises from the use of three *p* orbitals and one *s* orbital: the square arrangement involves the use of one *s*, two *p*, and one *d* orbital. This is what causes the four molecules of water in the hydrated cupric ion to have this arrangement.

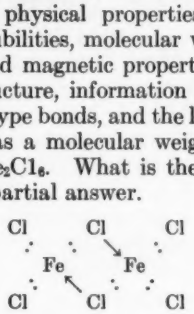
The table shows a summary of these arrangements.

Number of covalences	*Electronic orbitals involved*	*Spacial arrangement*	*Examples*
2	*s*, *p*	linear	CO_2
	d, *p*	linear	
3	*s*, *p*, *p*	trigonal plane	BCl_2, CO_3^{--}
	p, *p*, *p*	trigonal pyramid	NH_3, PH_3
	d, *s*, *p*	unsymmetrical plane	
4	*s*, *p*, *p*, *p*	tetrahedral	CH_4, $SnCl_4$
	d, *s*, *p*, *p*	square plane	$[Cu(H_2O)_4]^{++}$
5	*d*, *s*, *p*, *p*, *p*	bipyramid	PF_5, $MoCl_5$
	d, *d*, *s*, *p*, *p*	square pyramid	IF_5
6	*d*, *d*, *s*, *p*, *p*	octahedron	SF_6, $[PtCl_6]^{--}$
8	*d*, *d*, *d*, *d*, *s*, *p*, *p*, *p*	dodecahedron	$[Mo(CN)_8]^{-4}$

The electrons may be promoted from a lower shell to accomplish the necessary electronic configuration.

The methods used for detecting the structure are both physical and chemical and are too complicated

for examination at this time. The classical methods of determining the physical properties—melting point, boiling point, solubilities, molecular weights, electrical conductivities, and magnetic properties—give clues to differences in structure, information as to presence of covalent or ionic type bonds, and the kind of symmetry. Ferric chloride has a molecular weight corresponding to the formula Fe_2Cl_6. What is the structure? The diagram gives a partial answer.



The important point to be emphasized is that the chemical and physical properties depend upon the structure, and a satisfactory explanation for many seeming irregularities in behavior can only be explained by reference to the structure of the molecules or of the crystal. It is recommended that in high-school and freshman courses more attention be given to simple cases, such as the anomalous properties of water and ammonia, to show the strong influence that the structural factors have.

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To the Editor:

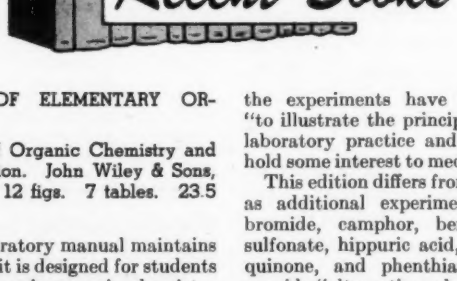
Dr. E. S. Cook (J. CHEM. EDUC., **28,** 597 (1951)) has made a stimulating contribution to the discussion of research in liberal arts colleges. For those of us who have been wrestling intimately with the problem this objective treatment is heartening.

The fact remains, however, that those engaged in teaching in liberal arts colleges are themselves slow in improving the situation. A check of *Chemical Abstracts* for the last 18 months (July, 1950 to December, 1951) to determine the volume of research produced in the South discloses that liberal arts colleges remain in the class of "one-percenters." The scores of such institutions in 15 southern states account for only 35 of the 3168 articles abstracted by this key to the world's chemical literature.

This latest survey broadens the "extremely narrow" basis for estimating the productive capacity of liberal arts colleges, but it does not brighten the picture.

JOHN R. SAMPEY

FURMAN UNIVERSITY
GREENVILLE, SOUTH CAROLINA



• LABORATORY MANUAL OF ELEMENTARY ORGANIC CHEMISTRY

George Holmes Richter, **Professor of Organic Chemistry and Dean, The Rice Institute. Second edition. John Wiley & Sons, Inc., New York, 1951. ix + 146 pp. 12 figs. 7 tables. 23.5 × 15.5 cm. $1.80.**

THE revision of this well-known laboratory manual maintains the objectives of its predecessor in that it is designed for students "required to take an elementary course in organic chemistry but specializing in the biological sciences." Accordingly, the experiments have been selected by Professor Richter "to illustrate the principal reactions and techniques of organic laboratory practice and their applications to products which hold some interest to medicine."

This edition differs from its excellent predecessor in containing as additional experiments the preparation of trimethylene bromide, camphor, benzoic acid, DDT, sodium benzenesulfonate, hippuric acid, chlorobenzene, 2-methyl-1,4-naphthoquinone, and phenthiazine. These have been included to provide "alternative selections or intermediates in other preparations." In addition, the procedures in other experiments

have been modified "to simplify the technique or improve the yield." A pedagogically valuable innovation is the pertinent questions at the end of each experiment.

The reviewer tested several of these experiments by assigning them to premedical students in his organic chemistry course. These students found the directions clear and obtained the expected quantities of products of good quality. The experiments in this manual are judiciously selected from the point of view of illustrating the principles of organic chemistry. The simple and "warm" style of the author reflects considerable experience in teaching this subject.

This manual will be welcomed by those teachers of organic chemistry who favor an introductory group of experiments on the principal laboratory techniques. It will also please those who prefer functional experiments inasmuch as each of these technique experiments employs compounds of some biological interest, *e. g.*, steam distillation of eugenol from cloves, melting point of acetanilide and antipyrine, and extraction of trimyristin from nutmeg. Another feature which should prove stimulating to students is the brief discussion of the applications of the compounds which is appended to each synthesis.

Inasmuch as micro and semimicro techniques have become increasingly important in the biological sciences several experiments should be included to illustrate these methods. Another suggestion is the inclusion in the student's laboratory report of a flow sheet for each synthesis experiment as a device for teaching both side-reactions and the principles involved in purifying organic compounds.

With the exception of these few minor omissions this laboratory manual represents a high standard of pedagogical achievement and is enthusiastically recommended by the reviewer.

JACOB G. SHAREFKIN

Brooklyn College
Brooklyn, New York

• THE PLANT GLYCOSIDES

R. J. McIlroy, Professor of Chemistry, University College, Ibadan, Nigeria, West Africa. Edward Arnold & Co., London, 1951. American Distributors: Longmans, Green and Co., New York. 138 pp. 2 tables. 14.5 × 22.5 cm. $3.25.

This book is a brief survey of most of the important plant glycosides, arranged, so far as is possible, according to the chemical nature of the aglycon. It should prove to be a useful reference source, especially in view of the fact that it carries the survey to a much more recent point than existing monographs—in particular Klein's classic "Handbuch."

The book would have been of greater value had the author included such details as the molecular formulas, particularly of those glycosides the constitutions of which have not yet been elucidated; and the more important properties—especially color, solubilities, and characteristic derivatives—of the compounds mentioned. Such information would not add greatly to the bulk of what was clearly intended to be a short monograph.

Attention must be called to numerous errors of fact, especially in the discussion of flavones, which are not "soluble in dilute mineral acids," are not all precipitated by lead acetate and whose pyrilium salts do not have the structure given for them. Certain minor errors in nomenclature (syringenin), typography (choline, "Agare" for "Agave"), and an occasional uncritical reporting of, for example, color reactions which do not have the specificity attributed to them, and of the physiological effects of "vitamin P," are also to be noted.

The book will be a useful one for those who are interested in the chemical substances found in plants, and its brevity will probably not detract greatly from its value because the literature references are sufficiently numerous and well-selected to facilitate access to the original publications. It is to be hoped that the author will present revised editions from time to time and thereby offer a focal point for this rather diffuse area of investigation.

T. A. GEISSMAN

University of California
Los Angeles, California

• CHAMBER'S DICTIONARY OF SCIENTISTS

A. V. Howard. E. P. Dutton and Co., Inc., New York, 1951. v + 500 numbered half-page columns. 70 portraits. 14.5 × 23 cm. $4.

The compiler of a volume of this kind is inevitably faced with the problem of whom to include. There will never be unanimity of opinion on this point, particularly as regards the omissions. The choices here seem eminently satisfactory.

The biological and physical sciences are both well represented. More than 300 chemists are included among the 1300 biographical sketches. The time span extends from the pre-Christian era to the present; many living scientists are on this honor roll. A table of the Nobel Laureates (1901–50) in chemistry, physics, and medicine occupies the back end-papers.

The sketches are constructed along the general pattern: full name; country of birth or adoption or both; special field; date of birth (in full or year only), date of death; training; principal positions occupied; précis of the principal research fields and achievements. The data are accurate and the condensed statements of the principal accomplishments are well done.

The book is full of valuable information in a readily accessible form. The reviewer expects to keep this handy reference volume within ready reach and consult it frequently.

RALPH E. OESPER

University of Cincinnati
Cincinnati, Ohio

• ASPECTS OF THE CONSTITUTION OF MINERAL OILS

K. Van Nes and *H. A. Van Westen,* Koninklijke–Shell-Laboratory, Amsterdam. Elsevier Publishing Co., Inc., New York, 1951. xxiv + 484 pp. 108 figs. 110 tables. 15 × 23 cm. $9.

This book brings together in one volume our present knowledge concerning methods for characterizing high-boiling fractions of crude oils. A major portion of it discusses a new technique for determining the carbon distribution, (percentage carbon in aromatic, naphthenic, and paraffinic structures), and ring content (average number of naphthenic and aromatic rings per molecule) in heavy oils.

Within the past few years refinery chemists have solved the problem of isolating the individual hydrocarbons in the gasoline range. Attention has turned to the kerosene, gas oil, and lubricating oil ranges where the problem is considerably more difficult due to the large number of components. As the authors point out, "The evident inextricability of heavier oil fractions made it necessary to devise methods for estimating the chemical composition of hydrocarbon mixtures, without complete separation." The methods of analysis presented in this book are based mainly on relations between various physical constants of the crude oil fractions. Only a brief mention is made of spectroscopic methods, such as ultraviolet absorption, for estimating the components of heavier oil fractions.

The first section of the book contains a review of our present knowledge concerning the origin and evolution of petroleum. The purpose of this is to determine to what extent data on the genesis of petroleum can contribute to a better understanding of its composition. This section is followed by an excellent chapter of the properties of hydrocarbons, and methods for their separation, characterization, and identification. Various formulas for computing the physical constants of pure hydro-

carbons are presented, as well as numerous characterization factors for determining the composition of complex mixtures. This chapter alone contains nearly 300 references.

The second half of the book is devoted to methods and applications of structural group analysis, specifically the estimation of carbon distribution and ring content in high-boiling oil fractions. The limitations of the original Waterman ring analysis are discussed and followed by a description of the development and testing of the new *n-d-M* (refractive index, density, molecular weight) method. The authors are to be complimented for including a fairly thorough discussion of other methods of structural group analysis such as those of Deanesly and Carleton, Lipkin, Martin, and Kurtz. Approximately 50 pages are devoted to a description and comparison of the different methods for structural group analysis. The limitations of all methods, including those of the authors, are covered fully. An interesting point in this discussion is the fact that it is incorrect to assume that aromatic oil fractions contain only minor amounts of naphthene rings. Present data indicate that mixed naphthenic-aromatic hydrocarbons are far more common than aromatics without naphthene rings in the heavier fractions of crude oils.

The material is presented in a very readable manner. Although some of the equations are hard to follow, there are numerous graphs which give the reader a quick visual interpretation of the various relationships between physical properties and composition. Application of the *n-d-M* method to specific analytical problems is simplified through the use of nomographs. The book should be a must for any chemist concerned with the analysis of high-boiling hydrocarbon mixtures.

JOHN M. HUNT

THE CARTER OIL COMPANY
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● BIOCHEMISTRY AND PHYSIOLOGY OF PROTOZOA, VOLUME I

Edited by *André Lwoff*, Head of the Department of Microbial Physiology, Pasteur Institute, Paris. Academic Press, Inc., New York, 1951. x + 434 pp. 59 figs. 44 tables. 15.5 × 24 cm. $8.80.

THE introductory chapter by A. Lwoff, and the second chapter by Hutner and Provasoli, deal to a considerable extent with the phytoflagellates. Theories on the phylogeny of the Protozoa *sensu stricto* and their probable relationships to the phytoflagellates and/or red bacteria are evaluated. While some of the phytoflagellates, like the green metaphytes, are nourished from inorganic sources and produce starch or paramylon through photosynthesis many are "acetate flagellates," unable to derive nourishment from glucose. Some are strict osmotrophs; others are phagotrophs. Some require growth factors such as thiamine and vitamin B_{12}. Excellent material for experimental work on photosynthesis is furnished by species that can live, with loss of chloroplasts, in complete darkness or in the presence of streptomycin. Synthesis and degradation of starch appear to be effected by enzymes of the Embden-Meyerhof-Parnas scheme. Experiments are suggested on factors of permeability of dissolved substances which have a bearing on the nutrition of all osmotrophs including the Protozoa *sensu stricto*. Experimental work is suggested throughout, since few factors of metabolism are known.

The first of the two chapters by M. Lwoff is an excellent review of the pioneer work of A. and M. Lwoff on certain Trypanosomidae, particularly on *Leishmania*, *Strigomonas*, and *Trypanosoma cruzi*. Excepting some strigomonads which do not appear to need exogenous growth factors, protoporphyrin, ascorbic acid, and B vitamins, particularly thiamine, are required. Nitrogenous foods may be derived from serum and/or peptones. A paper by Tobie and Rees which was overlooked shows that *T. cruzi* may be grown in dialysates of blood-agar.

The author attributes the bacteria-free cultivation of trichomonad flagellates to the use of antibiotics, overlooking the fact that most of the pure cultures were obtained by other methods, including microisolation. Excellent studies by Cailleau, Trussel, Johnson, and Morgan are reported in considerable detail. The limited literature on the growth requirements of parasitic amoebae, particularly *Endamoeba histolytica*, is admirably summarized and evaluated. Only limited information on these amoebae is obtainable because no pure cultures have been produced.

The chapter by von Brand dealing almost entirely with Trypanosomidae is subdivided into four sections: metabolism proper, pathological physiology of infections, physiological basis of chemotherapy, and physiological basis of drug resistance. A thoroughgoing review and evaluation of 269 research papers is presented. Most trypanosomes are active organisms relying principally on sugar as source of energy. A continuous supply is needed since little if any storage of glycogen occurs. Trypanosomes may be classified into two great groups of which the first, including species of *Leishmania* and several species of *Trypanosoma*, the so-called *lewisi* group, has heavy metal catalysts that are lacking in the second group which includes the pathogenic African species and *T. equiperdum*. A great handicap to studies on metabolism is that none of the blood-stream forms have thus far been cultivated *in vitro*. The section on pathological physiology is largely a discussion of theories since practically nothing is known about factors of pathogenicity. The author shows, however, that no simple explanations such as sugar depletion of the host, asphyxiation, etc., are adequate. The toxin theory is plausible but thus far toxins have not been demonstrated experimentally. The mode of action of most trypanocidal agents is explained in the section III in so far as valid data have been obtained. Of the many theories discussed in the fourth section on the physiological basis of drug resistance, the author favors the mutation theory.

The chapter by McKee on the biochemistry of *Plasmodium* and influence of antimalarials is a tribute to the outstanding collaborative researches of "teams" including biochemists, physicists, physiologists, bacteriologists, and protozoologists, at such centers as Harvard, the University of Chicago, Johns Hopkins University, and the National Institutes of Health. In this connection A. Lwoff comments in the introductory chapter that "close cooperation between protozoologists, botanists, parasitologists, and biochemists is a necessary condition for such progress." McKee stated that "plasmodia are able to glycolyze and to oxidize carbohydrate, to cleave hemoglobin, and to synthesize new proteins of a wide variety, and to produce large amounts of lipids." An outstanding accomplishment of the Harvard team was the cultivation of *Plasmodium* through enough generations to permit the *in vitro* testing of antimalarials. This result was accomplished through chemical studies of blood and plasma, before, during, and after infection, and through other studies which led to a physiologically balanced culture fluid which has been found useful also in the cultivation of other species of protozoa. Although many data are presented on the action of antimalarials the author states that there is as yet no detailed explanation for their selective action upon the plasmodia.

The chapter by Kidder and Dewey on the biochemistry of ciliates in pure culture shows that most of the essential inorganic nutrients of the aerobic ciliate *Tetrahymena gelei* have been found, as well as the essential amino acids and growth factors. Excepting "protogen," a concentrate from liver, the organism occupies a distinctive place in being the only protozoan that has been grown in a synthetic medium.

The book is well printed, remarkably free of typographical errors, and contains a remarkable number of references to pertinent literature, as well as a subject and author index.

CHARLES W. REES

SILVER SPRING, MARYLAND

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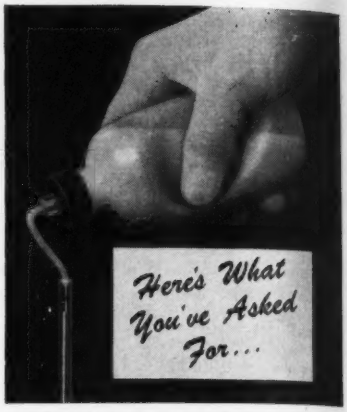

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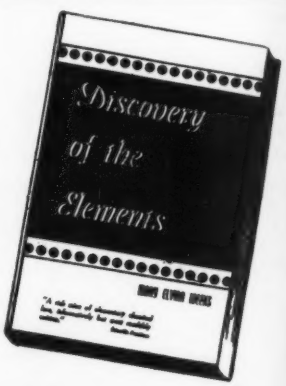

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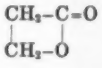

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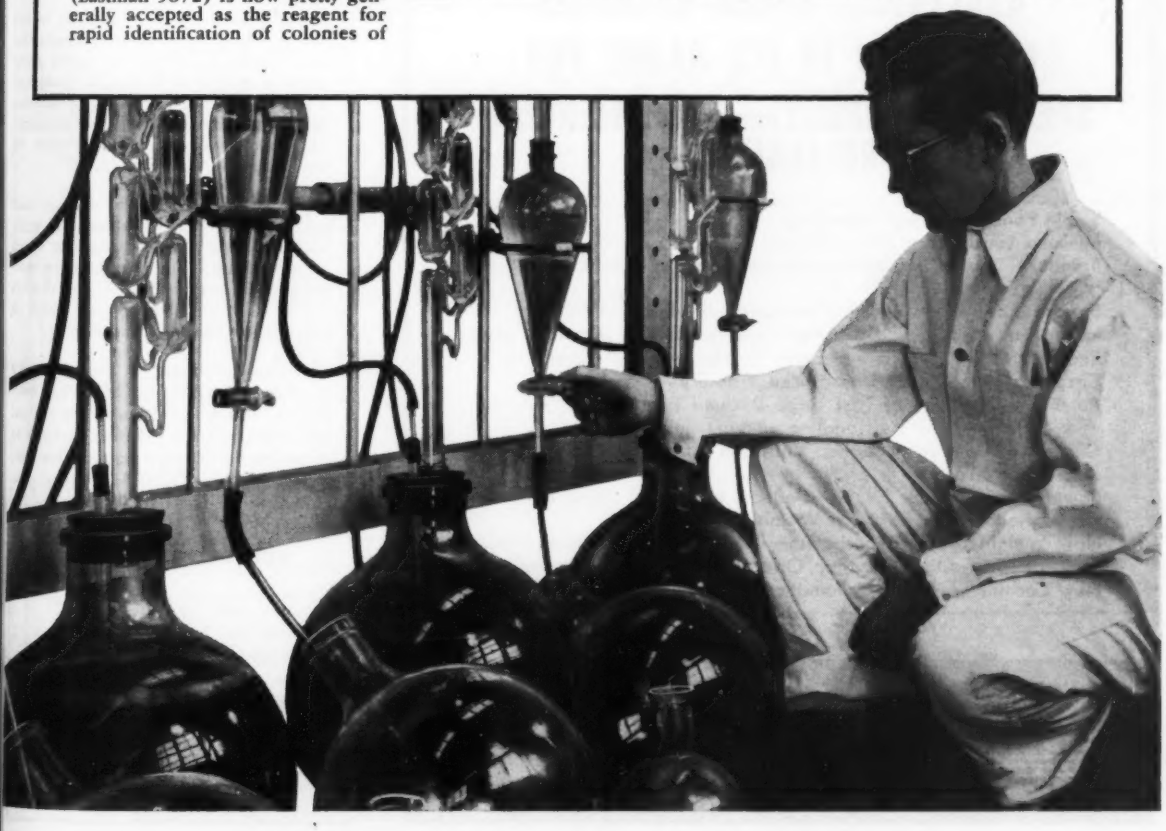

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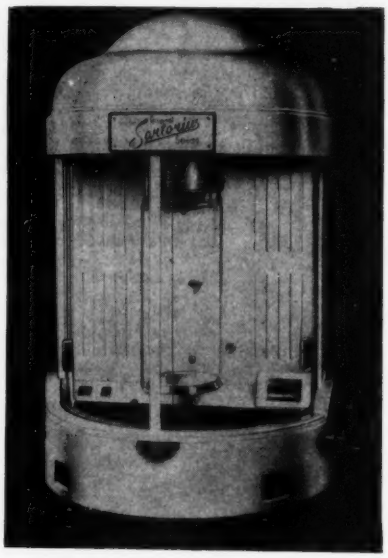

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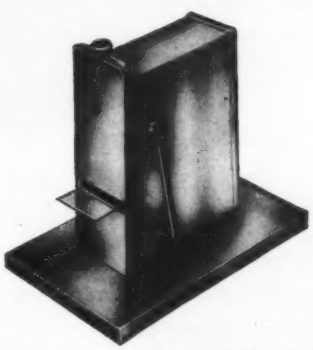

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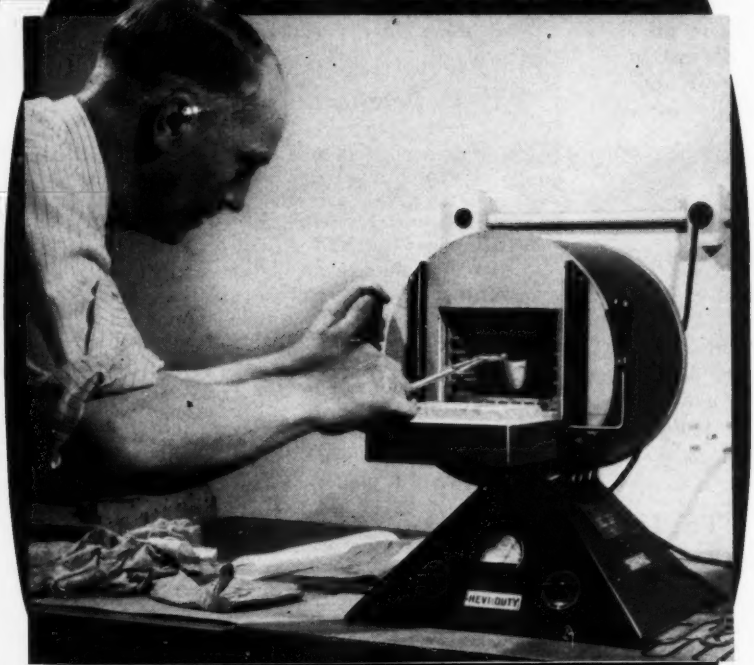

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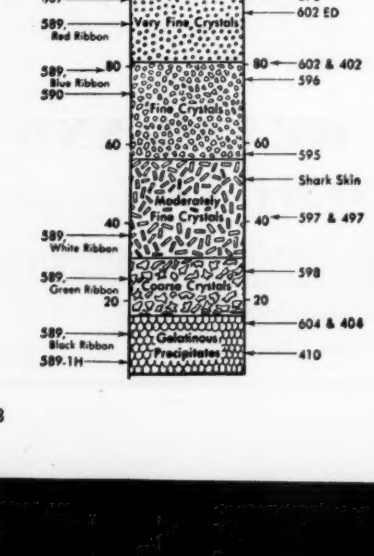

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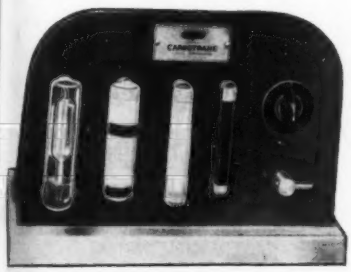

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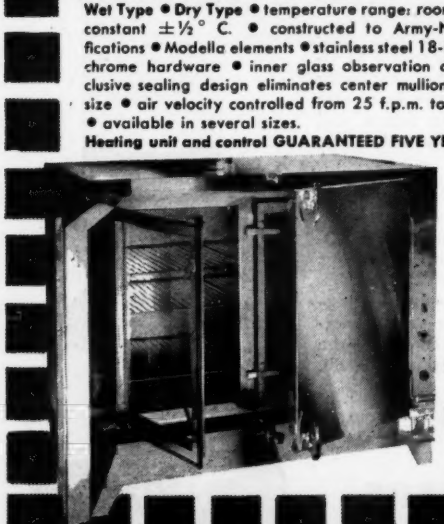

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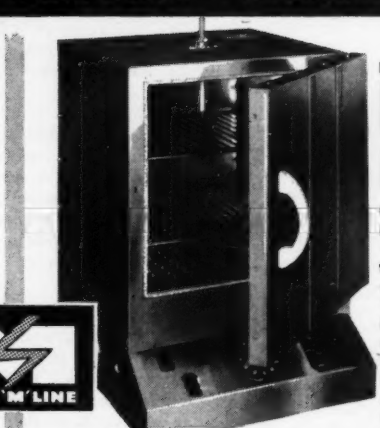

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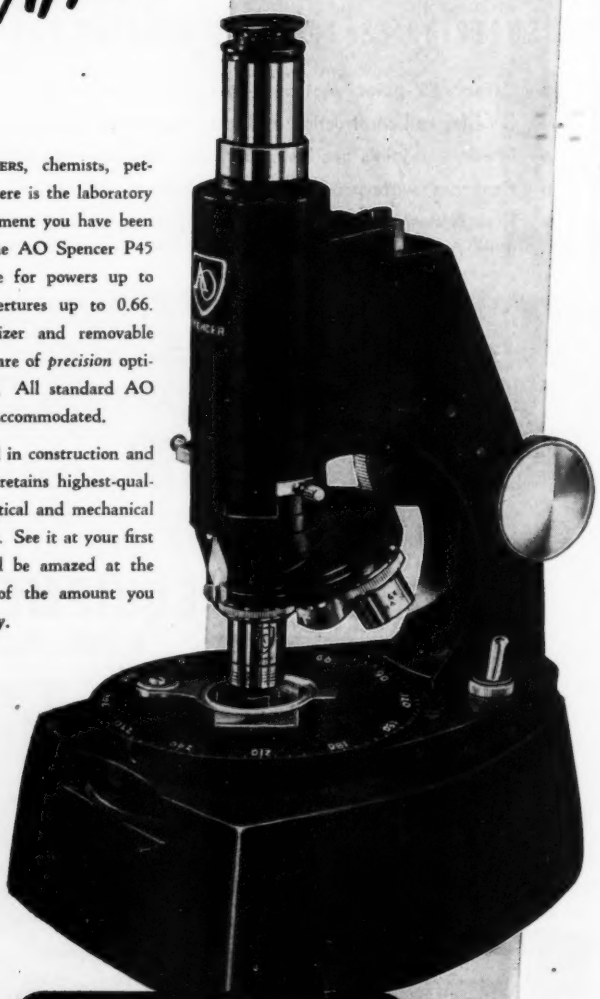

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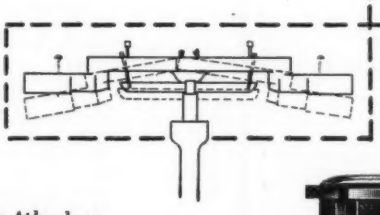

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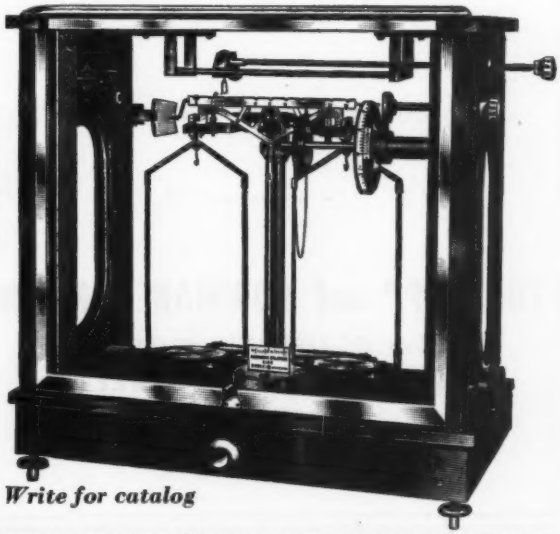

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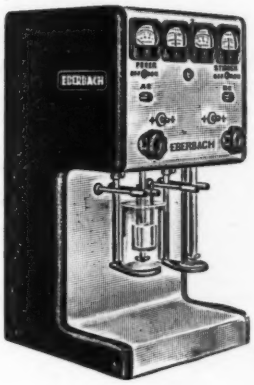

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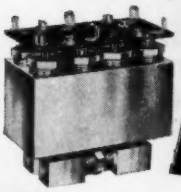

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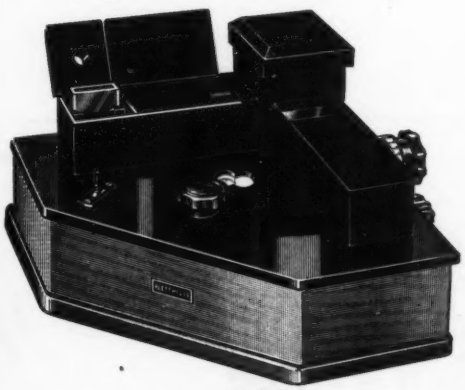

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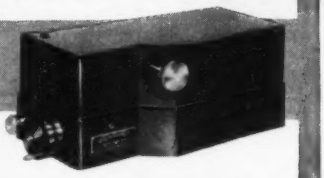

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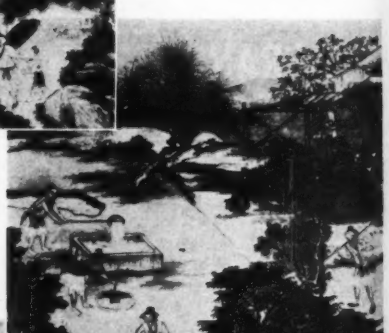

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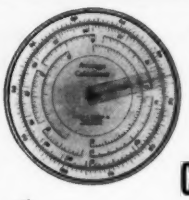

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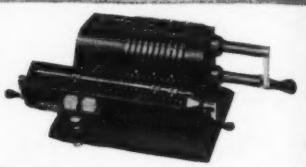

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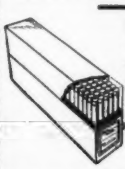

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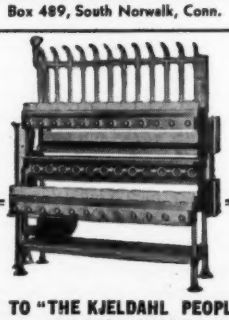

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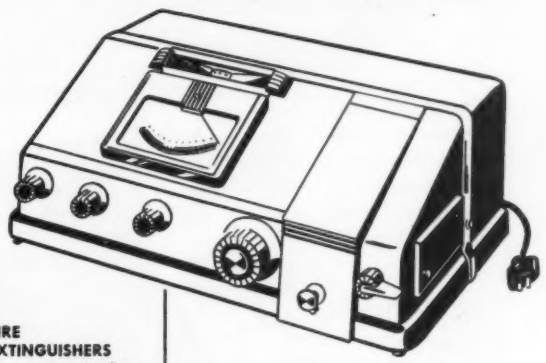

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CAP
The screw cap, introduced for the packaging of chemicals by Mallinckrodt in the early 20's.

PROTECTIVE SEAL
Assures the consuming chemist of factory-packaged chemicals—of original purity.

LABEL
White on black for unmistakable readability—leaves no room for reading errors. For your protection hazardous chemical labels have precautionary information on a side panel.

BOTTLE
Shaped for easy handling. Removal of chemicals made convenient by the wide mouth and sloping shoulder.

SIDE PANEL
Introduced by Mallinckrodt to avoid label congestion and to give greater legibility to the important maximum limits of impurities.

$BaCl_2+2H_2O$ M.W. 244.31

Meets A. C. S. Specifications

Maximum Limits of Impurities

Alkali Salts (as Sulfates)	0.050 %
Calcium and Strontium Salts (as Sulfates)	0.15 %
Insoluble Matter	0.010 %
Iron (Fe)	0.0002 %
Other Heavy Metals (as Pb)	0.0005 %
Nitrate and Chlorate (as NO_3)	0.005 %

YAD-1

Control

The cap, bottle and label of this package are the result of experience in supplying chemists with Analytical Reagents since 1912.

Mallinckrodt AR's are also available in bulk containers which have been developed with equal care and concern.

The extreme purity of AR's must be protected! Packaging is part of the product.

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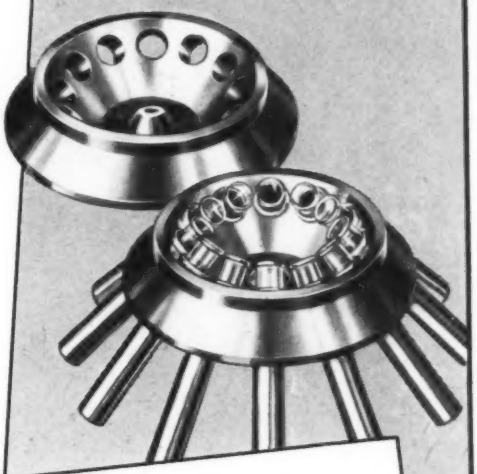

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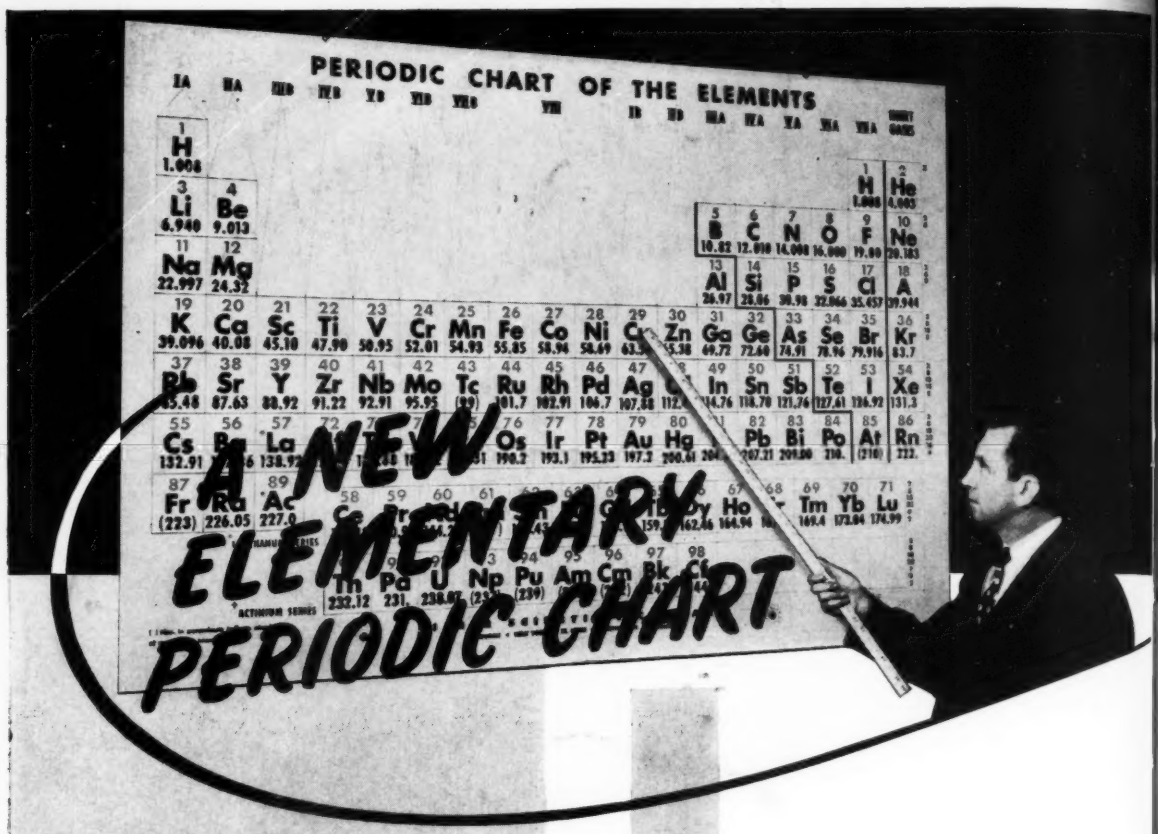

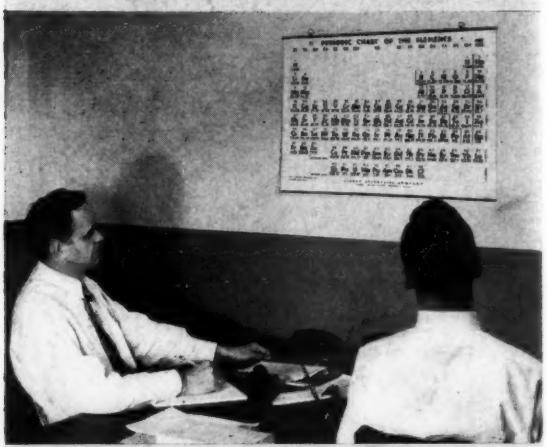

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